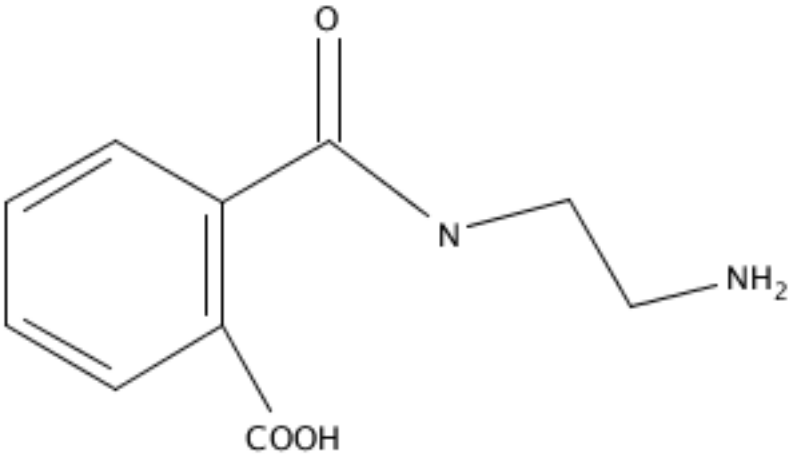
Task History

Task Began April 27, 2018 06:52 AM

Explore substances by SUBSTRUCTURE ID(3)



Answer Type: *Substances*
Result Count: *183*

Retrieve reference information in 1 substance (ID 6)

From ID: 3
Answer Type: *References*
Result Count: *150*

1. Methods and compounds for the treatment of bone loss and/or pain associated with activation of osteoclasts using a peptidylarginine deiminase inhibitor

By Catrina, Anca; Svensson, Camilla; Klareskog, Lars; Malmstrom, Vivianne
From U.S. Pat. Appl. Publ. (2017), US 20170105971 A1 20170420, Language: English, Database: CAPLUS

Bone loss and/or pain assocd. with an elevated activation of osteoclasts is prevented, treated and/or alleviated by the administration of an effective amt. of a compd. capable of inhibiting the activity of a peptidylarginine deiminase (PAD) enzyme. Methods and compds. for this use are disclosed, as well as diagnostic methods, kits, and a method for identifying compds. effective to prevent, treat and/or alleviate bone loss and/or pain.

~0 Citings

2. Methods and compounds for the alleviation and/or prevention of bone loss using a peptidylarginine deiminase inhibitor

By Catrina, Anca; Svensson, Camilla; Klareskog, Lars; Malmstroem, Vivianne
From PCT Int. Appl. (2017), WO 2017007405 A1 20170112, Language: English, Database: CAPLUS

Bone loss and/or pain assocd. with an elevated activation of osteoclasts is prevented, treated and/or alleviated by the administration of an effective amt. of a compd. capable of inhibiting the activity of peptidylarginine deiminase (PAD) enzymes. Methods and compds. for this use are disclosed, as well as diagnostic methods and kits.

~0 Citings

3. Protein arginine deiminase inhibitors as novel therapeutics for rheumatoid arthritis and cancer

By Thompson, Paul R.; Causey, Corey P.
From U.S. (2014), US 8921595 B2 20141230, Language: English, Database: CAPLUS

In accordance with certain embodiments of the present disclosure, a self-assembling biodegradable nanoparticle is provided. The nanoparticle includes Cys-Val-Val-Val-Val-Val-Val-Lys-Lys conjugated with a synthetic polymer and has a diam. of from about 50 nm to about 150 nm.

~0 Citings

4. Treatment of sepsis and septic shock by administering an agent that can reduce the level of circulating citrullated histones

By Alam, Hasan B.; Li, Yongqing
From PCT Int. Appl. (2015), WO 2015116896 A1 20150806, Language: English, Database: CAPLUS

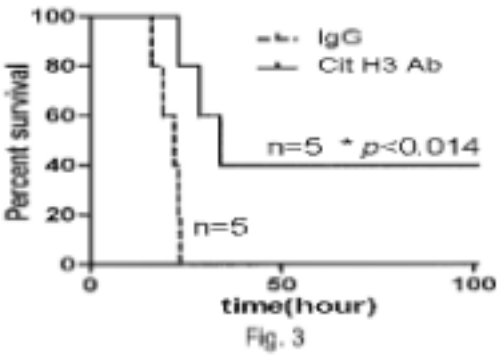


Fig. 3

The technol. described herein is directed to the treatment of sepsis and/or septic shock by, e.g. administering an agent that can reduce the level of circulating citrullated histones.

~0 Citings

5. Identification and characterization of related substances in pomalidomide by hyphenated LC-MS techniques

By Lu, Ping; Wang, Lei; Song, Min; Hang, Tai-jun
From Journal of Pharmaceutical and Biomedical Analysis (2015), 114, 159-167. Language: English, Database: CAPLUS, DOI:10.1016/j.jpba.2015.05.018

The current study dealt with the sepn., identification and characterization of related substances in pomalidomide by hyphenated techniques. Complete sepn. was obtained with an Inertsil ODS-SP column (250 mm × 4.6 mm, 5 μm) by linear gradient elution using a mobile phase consisting of 0.1% formic acid soln. and acetonitrile. They were characterized by hyphenated chromatog. techniques with the accurate mass detn. using high resoln. LC-TOF-MS methods as well as the product MS spectra detn. and elucidation. The degrdn. behaviors of pomalidomide under ICH prescribed stress conditions were also conducted. Pomalidomide was found to be labile to degrade under acid, alk., oxidative and thermal stress conditions, while it was relatively stable to photolytic stress. 13 related substances were detected and identified to be 10 degrdn. products and three process related substances. The hyphenated LC-MS method with high resoln. accurate mass detn. facilitated the qual. anal. of the unknown compds. than that of the conventional HPLC-UV. The related compds. identified are valuable for pomalidomide manufg. process optimization and quality control.

~2 Citings

6. Model of complex chiral drug metabolic systems and numerical simulation of the remaining chirality toward analysis of dynamical pharmacological activity

By Ogino, Yoshiyuki; Asahi, Toru
From Journal of Theoretical Biology (2015), 373, 117-131. Language: English, Database: CAPLUS, DOI:10.1016/j.jtbi.2015.03.011

In this study, systems of complicated pathways involved in chiral drug metab. were investigated. The development of chiral drugs resulted in significant improvement in the remedies available for the treatment of various severe sicknesses. Enantiopure drugs undergo various biol. transformations that involve chiral inversion and thus result in the generation of multiple enantiomeric metabolites. Identification of the specific active substances detg. a given drug's efficacy among such a mixt. of different metabolites remains a challenge. To comprehend this complexity, we constructed a math. model representing the complicated metabolic pathways simultaneously involving chiral inversion. Moreover, this model is applied to the metab. of thalidomide, which has recently been revived as a potentially effective prescription drug for a no. of intractable diseases. The numerical simulation results indicate that retained chirality in the metabolites reflects the original chirality of the unmetabolized drug, and a higher level of enantiomeric purity is preserved during spontaneous degrdn. In addn., chirality remaining after equilibration is directly related to the rate const. not only for chiral inversion but also for generation and degrdn. Furthermore, the retention of chirality is quant. predictable using this combination of kinetic parameters. Our simulation results well explain the behavior of thalidomide in the practical biol. exptl. data. Therefore, this model promises a comprehensive understanding of dynamic metabolic systems involving chiral drugs that express multiple enantiospecific drug efficacies.

~0 Citings

7. Thalidomide

By Penner, Natasha
Edited By:Lee, Philip W
From Handbook of Metabolic Pathways of Xenobiotics (2014), 5, 2222-2224. Language: English, Database: CAPLUS

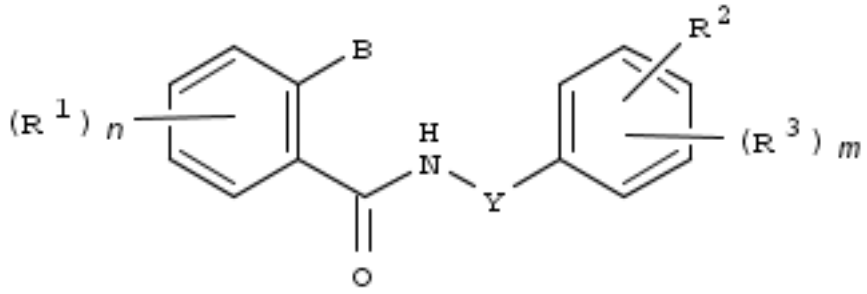
This article describes the hydrolytic degrdn. and primary metabolic pathway of thalidomide in animals and human liver microsomes. Thalidomide is a sedative drug that was used to treat morning sickness and to aid sleep. The current use of thalidomide is to treat and prevent the debilitating and disfiguring skin sores caused by erythema nodosum leprosum, an inflammatory complication of leprosy and multiple myeloma (a form of bone marrow cancer). Urine samples from seven multiple myeloma patients at steady state levels of thalidomide therapy showed the presence of only three hydrolysis products and no hydroxylated metabolites was detected.

~0 Citings

8. Compounds inducing chondrogenesis and their preparation

By Schultz, Peter G.; Chatterjee, Arnab K.; Zhu, Shoutian; Payette, Joshua; Yoon, Hongchul; Yang, Baiyuan
From U.S. Pat. Appl. Publ. (2014), US 20140271955 A1 20140918, Language: English, Database: CAPLUS

The invention relates to compds. of formula I inducing chondrogenesis and their prepn. Compds. I, wherein R^1 is halo, alkyl, alkoxy, etc.; R^2 is halo, alkyl, haloalkyl, etc.; R^3 is H, CN, halo, etc.; n is 0-4; m is 1-4; B is substituted Ph, CH_2COOH, alkylcarbonyl, etc.; Y is a bond or alkyl; their pharmaceutically acceptable salts, solvates, polymorphs, prodrugs, esters, metabolites, N-oxides, stereoisomers, or isomers, are claimed. The invention compds. can be applied for the amelioration of arthritis or joint injuries by inducing mesenchymal stem cells into chondrocytes.



I

~0 Citings

9. Water mediated, environmentally friendly, step-wise, tandem & one-pot syntheses of 2-(1H-benzo[d]imidazole-2-yl)-N-arylbenzamides

By Reddy, Yervala Dathu; Ramana Reddy, Chittireddy Venkata; Dubey, Pramod Kumar
From RSC Advances (2014), 4(6), 2974-2979. Language: English, Database: CAPLUS, DOI:10.1039/C3RA44423F

Water mediated and environmentally friendly, step-wise, tandem & one-pot syntheses of 2-(1H-benzo[d]imidazole-2-yl)-N-arylbenzamide derivs. were developed by simply combining phthalic anhydride, anilines and phenylenediammonium dihydrogenphosphate. This reaction has an easy workup, provides excellent yields, and uses water as the solvent which is considered to be relatively environmentally benign.

~12 Citings

10. The protein arginine deiminases: Structure, function, inhibition, and disease

By Bicker, Kevin L.; Thompson, Paul R.
From Biopolymers (2013), 99(2), 155-163. Language: English, Database: CAPLUS, DOI:10.1002/bip.22127

A review. The post-translational modification of histones has significant effects on overall chromatin function. One such modification is citrullination, which is catalyzed by the protein arginine deiminases (PADs), a unique family of enzymes that catalyzes the hydrolysis of peptidyl-arginine to form peptidyl-citrulline on histones, fibrinogen, and other biol. relevant proteins. Over-expression and/or increased PAD activity is obsd. in several diseases, including rheumatoid arthritis, Alzheimer's disease, multiple sclerosis, lupus, Parkinson's disease, and cancer. This review discusses the important structural and mechanistic characteristics of the PADs, as well as recent investigations into the role of the PADs in increasing disease severity in RA and colitis and the importance of PAD activity in mediating neutrophil extracellular trap formation through chromatin de-condensation. Lastly, efforts to develop PAD inhibitors with excellent potency, selectivity and in vivo efficacy are discussed, highlighting the most promising inhibitors.

~40 Citings

11. D-Amino Acid-Based Protein Arginine Deiminase Inhibitors: Synthesis, Pharmacokinetics, and in Cellulo Efficacy

By Bicker, Kevin L.; Anguish, Lynne; Chumanevich, Alexander A.; Cameron, Michael D.; Cui, Xiangli; Witalison, Erin; Subramanian, Venkataraman; Zhang, Xuesen; Chumanevich, Alena P.; Hofseth, Lorne J.; et al
From ACS Medicinal Chemistry Letters (2012), 3(12), 1081-1085. Language: English, Database: CAPLUS, DOI:10.1021/ml300288d

The protein arginine deiminases (PADs) are known to play a crucial role in the onset and progression of multiple inflammatory diseases, including rheumatoid arthritis, inflammatory bowel disease, and cancer. However, it is not known how each of the five PAD isoenzymes contributes to disease pathogenesis. As such, potent, selective, and bioavailable PAD inhibitors will be useful chem. probes to elucidate the specific roles of each isoenzyme. Because D-amino amino acids often possess enhanced in cellulo stability, and perhaps unique selectivities, we synthesized a series of D-amino acid analogs of our pan-PAD inhibitor Cl-amidine, hypothesizing that this change would provide inhibitors with enhanced pharmacokinetic properties. Herein, we demonstrate that D-Cl-amidine and D-o-F-amidine are potent and highly selective inhibitors of PAD1. The pharmacokinetic properties of D-Cl-amidine were moderately improved over those of L-Cl-amidine, and this compd. exhibited similar cell killing in a PAD1 expressing, triple-neg. MDA-MB-231 breast cancer cell line. These inhibitors represent an important step in our efforts to develop stable, bioavailable, and highly selective inhibitors for all of the PAD isoenzymes.

~17 Citings

12. The development of N-α-(2-carboxyl)benzoyl-N5-(2-fluoro-1-iminoethyl)-L-ornithine amide (o-F-amidine) and N-α-(2-carboxyl)benzoyl-N5-(2-chloro-1-iminoethyl)-L-ornithine amide (o-Cl-amidine) as second generation protein arginine deiminase (PAD) inhibitors [Erratum to document cited in CA155:484468]

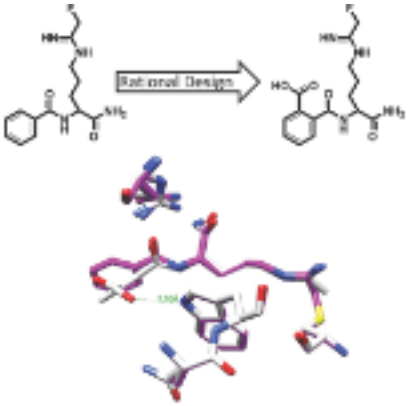
By Causey, Corey P.; Jones, Justin E.; Slack, Jessica L.; Kamei, Daisuke; Jones, Larry E., Jr.; Subramanian, Venkataraman; Knuckley, Bryan; Ebrahimi, Pedram; Chumanevich, Alexander A.; Luo, Yuan; et al
From Journal of Medicinal Chemistry (2011), 54(22), 7942. Language: English, Database: CAPLUS, DOI:10.1021/jm201411r

On page 6933, the PDB identification nos. contained errors; the cor. nos. are given.

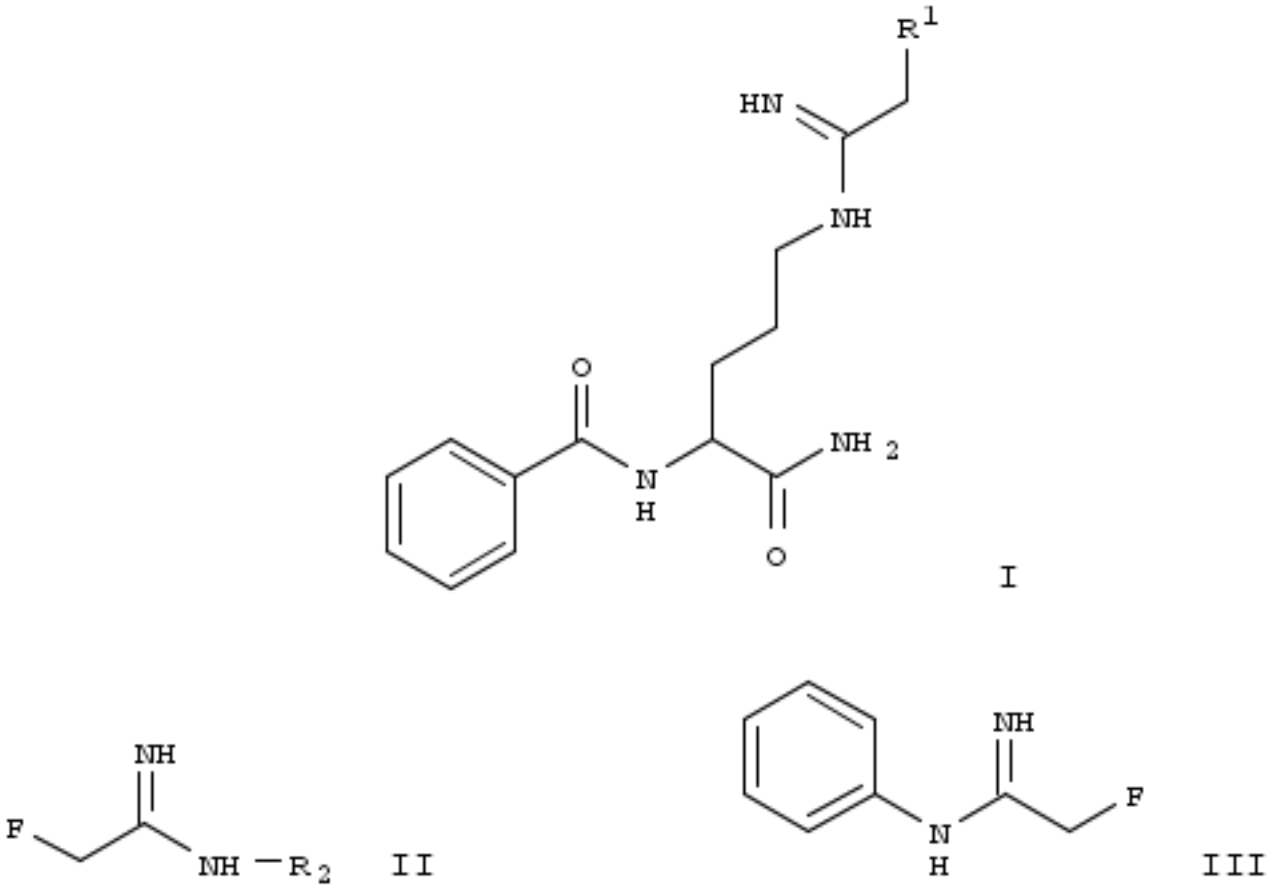
~0 Citings

13. The development of N-α-(2-carboxyl)benzoyl-N5-(2-fluoro-1-iminoethyl)-L-ornithine amide (o-F-amidine) and N-α-(2-carboxyl)benzoyl-N5-(2-chloro-1-iminoethyl)-L-ornithine amide (o-Cl-amidine) as second generation protein arginine deiminase (PAD) inhibitors

By Causey, Corey P.; Jones, Justin E.; Slack, Jessica L.; Kamei, Daisuke; Jones, Larry E.; Subramanian, Venkataraman; Knuckley, Bryan; Ebrahimi, Pedram; Chumanevich, Alexander A.; Luo, Yuan; et al
From Journal of Medicinal Chemistry (2011), 54(19), 6919-6935. Language: English, Database: CAPLUS, DOI:10.1021/jm2008985



Protein arginine deiminase (PAD) activity is upregulated in a no. of human diseases, including rheumatoid arthritis, ulcerative colitis, and cancer. These enzymes, there are five in humans (I) (R^1 = Cl, F), (II) (R^2 = H, CH_2CH_2Me) and (III), regulate gene transcription, cellular differentiation, and the innate immune response. Building on our successful generation of F- and Cl-amidine, which irreversibly inhibit all of the PADs, a structure-activity relationship was performed to develop second generation compds. with improved potency and selectivity. Incorporation of a carboxylate ortho to the backbone amide resulted in the identification of N-α-(2-carboxyl)benzoyl-N^5-(2-fluoro-1-iminoethyl)-L-ornithine amide (o-F-amidine) and N-α-(2-carboxyl)benzoyl-N^5-(2-chloro-1-iminoethyl)-L-ornithine amide (o-Cl-amidine), as PAD inactivators with improved potency (up to 65-fold) and selectivity (up to 25-fold). Relative to F- and Cl-amidine, the compds. also show enhanced potency in cellulo. As such, these compds. will be versatile chem. probes of PAD function.

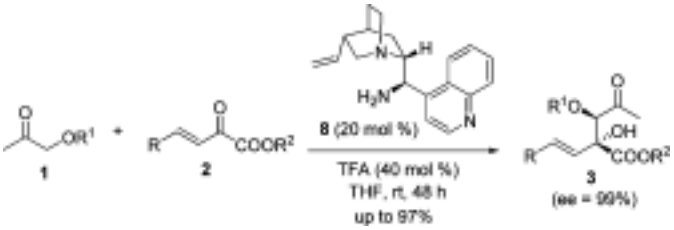


~33 Citings

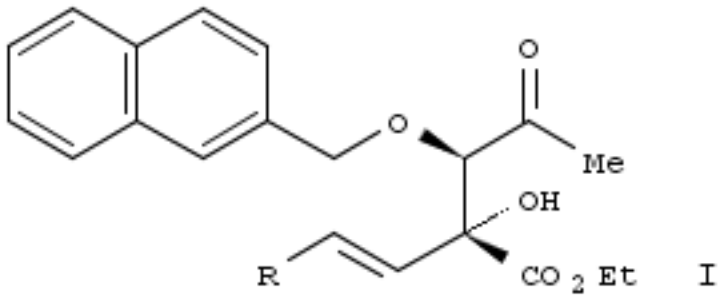
14. Organocatalytic Asymmetric Aldol Reaction of Hydroxyacetone with β,γ-Unsaturated α-Keto Esters: Facile Access to Chiral Tertiary Alcohols

By Liu, Chen; Dou, Xiaowei; Lu, Yixin

From Organic Letters (2011), 13(19), 5248-5251. Language: English, Database: CAPLUS, DOI:10.1021/ol2021274



An efficient direct asym. aldol reaction between hydroxyacetone and β,γ-unsatd. α-keto esters has been successfully developed. In the presence of 9-amino-9-deoxy-epi-cinchonine and trifluoroacetic acid, the direct aldol reaction of O-protected hydroxyacetone proceeded in a highly enantioselective manner, affording the desired adducts contg. a chiral tertiary alc., e.g. I (R = Ph, 3-BrC_6H_4, 2-MeC_6H_4, thiopene-3-yl, 3-pyridinyl), in high yields and with excellent enantioselectivities. The aldol products obtained are valuable precursors for the synthesis of 2-substituted glycerol derivs.



~36 Citings

15. Synthesis and insecticidal activities of phthalic diamides

By Wei, Wei; Zhu, Bingchun; Xing, Jiahua; Kong, Xiaolin; Tan, Chengxia; Li, Xiaonian
From Nongyao (2011), 50(4), 249-252. Language: Chinese, Database: CAPLUS

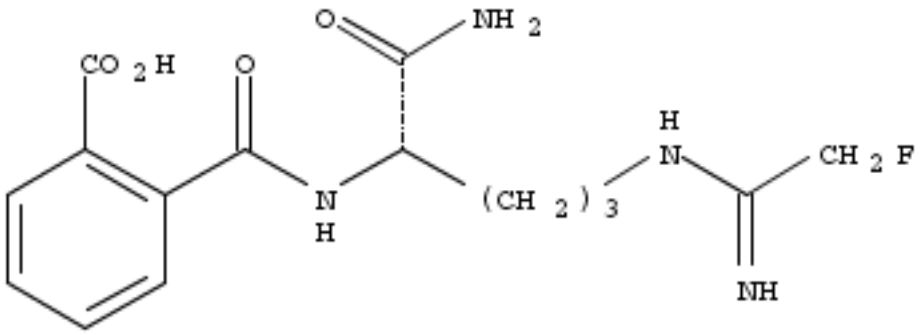
Thirteen novel phthalic diamides were synthesized from substituted phthalic anhydride, 2-amino-2-methylpropionitrile and heptafluoroisopropyl aniline. The chem. structures of the target compds. were confirmed by ^{1}H NMR, IR and MS. And the biol. activities of the target compds. were tested. The control effect of compds. 6a against Plutella xylostella, Helicoverpa armigera Hubner and Spodoptera frugiperda was 90, 100 and 75% at the concn. of 0.8 mg/L, against Helicoverpa armigera Hubner and Spodoptera frugiperda was 55 and 50% at the concn. of 0.16 mg/L, resp. These novel phthalic diamides showed high insecticidal activities against lepidopterous pests.

~0 Citings

16. Protein arginine deiminase inhibitors as novel therapeutics for rheumatoid arthritis and cancer

By Thompson, Paul R.; Causey, Corey
From PCT Int. Appl. (2011), WO 2011050357 A2 20110428, Language: English, Database: CAPLUS

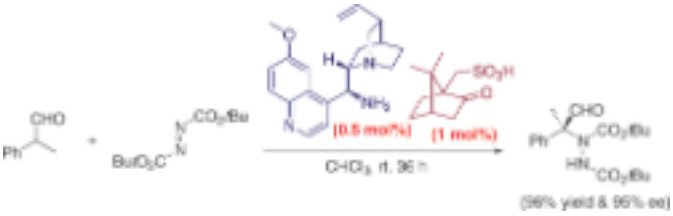
In accordance with certain embodiments of the present disclosure, a self-assembling biodegradable nanoparticle is provided. The nanoparticle includes Cys-Val-Val-Val-Val-Val-Val-Lys-Lys conjugated with a synthetic polymer and has a diam. of from about 50 nm to about 150 nm.



~2 Citings

17. Primary amine/CSA ion pair: a powerful catalytic system for the asymmetric enamine catalysis

By Liu, Chen; Zhu, Qiang; Huang, Kuo-Wei; Lu, Yixin
From Organic Letters (2011), 13(10), 2638-2641. Language: English, Database: CAPLUS, DOI:10.1021/ol200747x



A novel ion pair catalyst contg. a chiral counteranion can be readily derived by simply mixing cinchona alkaloid-derived diamine with chiral camphorsulfonic acid (CSA). A mixt. of 9-amino(9-deoxy)epi-quinine and (-)-CSA was found to be the best catalyst with matching chirality, enabling the direct amination of α-branched aldehydes to proceed in quant. yields and with nearly perfect enantioselectivities. A 0.5 mol % catalyst loading was sufficient to catalyze the reaction, and a gram scale enantioselective synthesis of biol. important α-Me phenylglycine has been successfully demonstrated.

~53 Citings

18. Hydrogenation products from dehydro-D-erythro- and dehydro-L-threo-ascorbic acids mono- and bishydrazones

By El Sekily, Mohamed A.
From Arabian Journal for Science and Engineering, Section A: Sciences (2008), 33(1A), 7-13. Language: English, Database: CAPLUS

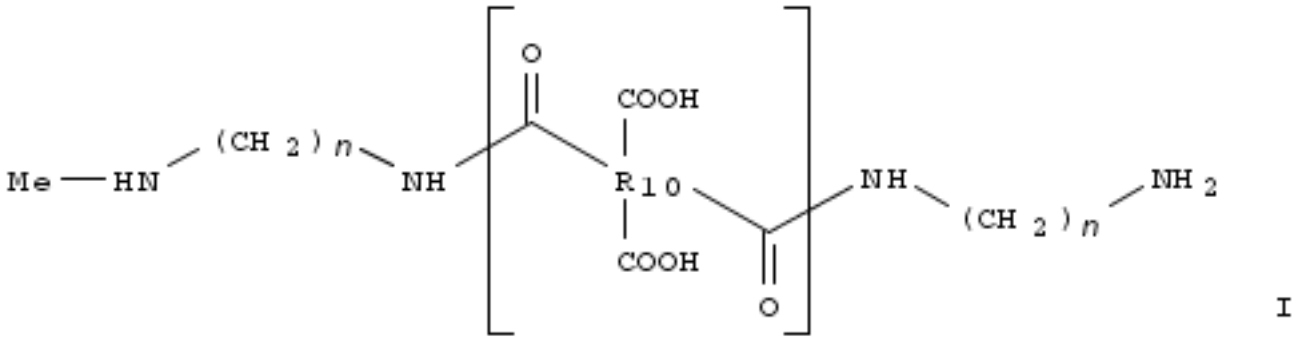
Reaction of dehydro-D-isoascorbic acid with benzoylhydrazine gave the bisbenzoylhydrazone which, upon hydrogenation in the presence of Pd/C catalyst, gave the 2,3-diamino-2,3-dideoxy-D-isoascorbic acid. Reaction of the latter compd. with different aldehydes afforded, the bicyclic imidazoline derivs., and with arylglyoxal, gave the bicyclic pyrazine derivs. The 2,3-diamino-2,3-dideoxy-L-ascorbic acid reacted with anhyd. HCOOH giving the monoformyl-deriv. and with glyoxal to give a bicyclic pyrazine deriv. $NaBH_4$ redn. of dehydro-L-ascorbic acid-2-p-chlorophenylhydrazone, gave the bicyclic 3,6-anhydro-deriv. which was hydrogenated in the presence of Pd/C to give the 2-amino-2-deoxy deriv.

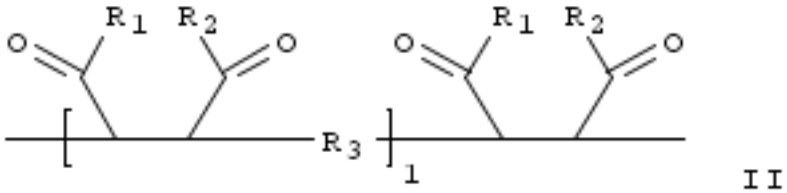
~1 Citing

19. Microarray substrate, method of analyzing a biomolecule using microarray substrate, and products comprising the microarray substrate

By Peak, Sang-Hyun; Park, Jong-Myeon; Yoo, Chang-Eun
From U.S. Pat. Appl. Publ. (2007), US 20070196836 A1 20070823, Language: English, Database: CAPLUS

Provided are a microarray substrate comprising a solid substrate coated with a chem. having a functional residue represented by Formula 1 or 2 below, a method of analyzing a biomol. using the microarray substrate, and a lab-on-a-chip comprising the microarray substrate: [I] [II] wherein n, the structure within brackets [ ], R_1, R_2, R_3, R_{10}, n and 1 are as defined in the specification.





~0 Citings

20. 2-[(2-Ammonioethyl)aminocarbonyl]benzoate hemihydrate

By Chellappa, D.; Chitraleka, S.; Sakthilatha, D.; Athimoolam, S.; Natarajan, S.
From Acta Crystallographica, Section E: Structure Reports Online (2007), 63(6), o2875-o2876. Language: English, Database: CAPLUS, DOI:10.1107/S1600536807021599

The amino acid-like structure of 2-[(2-ammonioethyl)aminocarbonyl]benzoate hemihydrate, $C_{10}H_{12}N_2O_3 \cdot 0.5H_2O$, consists of 2 zwitterionic residues and 1 H_2O mol. in the asym. unit. The carboxylate group is twisted from the plane of the attached benzene ring by angles of 35.6(4) and 36.2(5)° in the 2 residues. From the benzene ring, the side-chain conformations are trans/gauche-I/ gauche-II and trans/gauche-II/gauche-II. The crystal structure is stabilized by an intricate 3-dimensional H-bonding network. The amino and carboxylate groups are connected through intra- and intermol. H bonds, forming S(10), C(4), $C_2^1(4)$ and $C_3^3(8)$ motifs. The chains run along the a axis of the unit cell. Hydrophobic layers across z = 1/4 and 3/4 are sandwiched between the hydrophilic layers across z = 1/2 and 1. Crystallog. data are given.

~0 Citings

21. New synthetic route of R-orS-thalidomide

By Xiang, Dong; Cheng, Li; Wu, Xianxue; Wu, Chenglong; Yin, Hongmei; Wei, Yuquan
From Huaxue Yanjiu Yu Yingyong (2006), 18(11), 1349-1352. Language: Chinese, Database: CAPLUS

A novel synthetic route of thalidomide was found. We synthesized the target compds.(R-or S-thalidomide) through six procedures starting from glutamic acid in an overall yield of 61%. The intermediates and targets compds. were identified by IR, ^{1}H NMR, and elemental anal.

~1 Citing

22. Preparation of peptide nucleic acid conjugates

By Nielsen, Peter; Buchardt, Ole; Sonnechsen, Soren Holst; Lohse, Jesper; Egholm, Michael; Manoharan, Muthiah; Kiely, John; Griffith, Michael; Sprankle, Kelly
From U.S. (2007), US 7223833 B1 20070529, Language: English, Database: CAPLUS

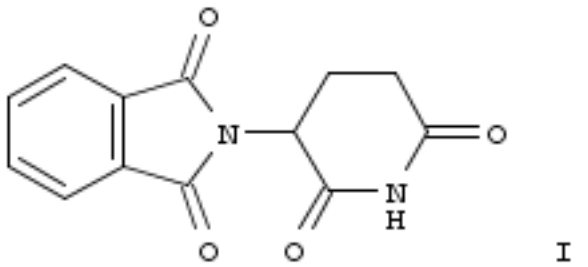
The invention is related to peptide nucleic acids (PNAs) which are functionalized to include covalently bound conjugates Q-[$E_mB_m(A_mL_m)D_mG_m]_m$-EB(AL)DI [m = 1-50; L, L_m = independently occurring nucleobases; E, E_m = CH_2; D, D_m = CH_2CH_2; G_m = NHCO in either orientation; each pair of A-A_m and B-B_m = N-CO-CH_2; I = NR^8R^9 or $NR^{10}COR^{11}$; R^8-R^{11} = independently H, alkyl, amino protecting group, a reporter ligand, an intercalator, a chelator, a peptide, a protein, a carbohydrate, a lipid, a steroid, a nucleoside, a nucleotide, a nucleotide diphosphate, a nucleotide triphosphate, an oligonucleotide, an oligonucleoside, a sol. polymer, a non-sol. polymer, a reporter enzyme, a reporter mol., a terpene, a phospholipid, a cell receptor binding mol., a water sol. vitamin, a lipid sol. vitamin, an RNA/DNA cleaving complex, a porphyrin, or a polymeric compd. selected from polymeric amines, polymeric glycols and polyethers; Q = CO_2H, CO_2R^8, or $CONR^8R^9$]. Thus, mixing PNA H-AHA-T-T-C-T-T-C-T-T-T-T-NH_2 (AHA = NH-$(CH_2)_5$COO) with folic acid in DMSO gave the folic acid-PNA conjugate. A PNA-acridine conjugate gave a 10-100 fold increase in its binding to dsDNA compared to the unmodified PNA. In a plasmid-DNA strand cleavage assay, the PNA-NTA (NTA nitrilotriacetic acid) conjugate, NTA-Lys-Lys-T-T-C-T-T-C-T-T-T-T-Lys-Lys-NH_2, effectively cleaved the DNA strand in the presence of Fe^{2+} and EDTA.

~13 Citings

23. Hydrolyzed metabolites of thalidomide: synthesis and TNF-α production-inhibitory activity

By Nakamura, Takanori; Noguchi, Tomomi; Miyachi, Hiroyuki; Hashimoto, Yuichi
From Chemical & Pharmaceutical Bulletin (2007), 55(4), 651-654. Language: English, Database: CAPLUS, DOI:10.1248/cpb.55.651

Putative hydrolyzed metabolites of thalidomide (I) were prepd. and characterized, and their inhibitory activity on tumor necrosis factor (TNF)-α prodn. in the human monocytic leukemia cell line THP-1 was evaluated. α-(2-Carboxybenzamido)glutarimide (II) was a more potent TNF-α prodn. inhibitor than I.



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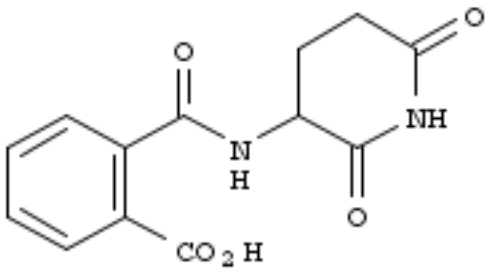


II

~10 Citings

24. Effect of positional isomerism on the abiotic degradation of pesticides: Case of m- and p-imazamethabenz-methyl

By Brigante, Marcello; Emmelin, Corinne; Ferronato, Corinne; Della Greca, Marina; Previtera, Lucio; Paisse, Jean Olivier; Chovelon, Jean-Marc
From Chemosphere (2007), 68(3), 464-471. Language: English, Database: CAPLUS, DOI:10.1016/j.chemosphere.2006.12.084

The effect of positional isomerism on chem. and photochem. degrdns. of the imazamethabenz-Me (IMBM) has been studied. IMBM is proposed in the form of a mixt. of the two positional isomers: meta and para. The development of an effective HPLC method (resoln. factor R = 1.3) allows us to follow the abiotic disappearance of the meta and para IMBM and the formation of their breakdown products. The abiotic degrdn. studies include chem. hydrolysis, as well as the direct and the indirect photodecompn. We used TiO_2, a well-known initiator of hydroxyl radicals, to highlight the role of ·OH in the indirect photodegrdn. This work confirms the different behaviors of positional isomers in the environment. The chem. or direct photochem. degrdn. is faster for the meta isomer than for the para. There is no influence of this type of isomerism on the indirect photochem. degrdn. The degrdn. products were tentatively identified by LC-MS, NMR and IR and a degrdn. pathway was proposed.

~12 Citings

25. Pyrolytic synthesis of heterocycles guided by mass spectral fragmentations

By Prakasam, T.; Srinivasan, P. S.; Arabindoo, Bhanumathi; Ramana, D. V.
From Indian Journal of Heterocyclic Chemistry (2006), 16(2), 143-146. Language: English, Database: CAPLUS

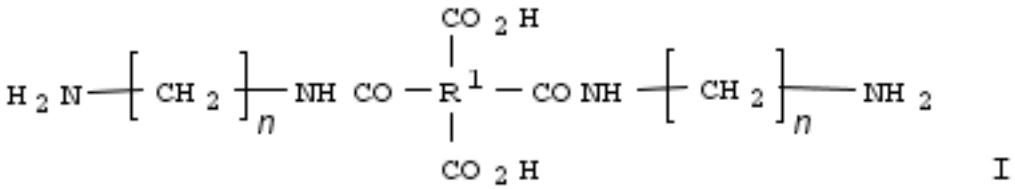
1,2-Benzoylenebenzimidazole, dibenzo[b,f]-1,4-(5H,12H)-diazocine-6,11-dione, 2-phenylbenzoxazole, 2-phenylbenzothiazole, N-(2-methylphenyl)phthalimide, N-(2-carboxyphenyl)phthalimide, N-phenylphthalimide, and thianthrene are prepd. through pyrolytic procedures from substituted phthalanilic acids and 2-nitrodiphenyl disulfide based on their mass spectral fragmentations. The pyrolytic expts. have been optimized for max. yields and purity of the products. These pyrolytic methods do not involve the use of hazardous chems.

~1 Citing

26. Capture and purification of nucleic acids using pH-responsive polymers containing amino and carboxyl groups

By Hwang, Kyu-Youn; Kim, Joon-Ho; Yoo, Chang-Eun; Lee, Hun-Joo; Lim, Hee-Kyun; Jeong, Sung-Young; Shim, Jeo-Young
From Eur. Pat. Appl. (2006), EP 1674570 A2 20060628, Language: English, Database: CAPLUS

Polymers for capture of nucleic acids (I, R^1 = tetracarboxylic dianhydride, n=1-10) that show pH-dependent binding of nucleic acids are described for use in purifn. These materials bind nucleic acids very strongly at an acid pH (~3) and show >99% release at a neutral or alk. pH. Amine activated glass slides were immersed in a soln. of 1,2,4,5-benzenetetracarboxylic acid dianhydride 100 mM in N-methyl-2-pyrrolidone (NMP) for 1h, washed with acetone, and dried. These slides were then immersed in a soln. of ethylenediamine in NMP for 1h, washed with acetone and dried. The slides bound DNA quant. at pH 3 (sodium acetate buffer, 0.15 M), elution of the DNA was complete at pH 7 (Tris buffer, 0.15 M.).



I

~1 Citing

27. Poly(amide imide)s and poly(amide imide) composite membranes by interfacial polymerization

By Buch, Premang R.; Mohan, D. Jagan; Reddy, Alamuru V. R.
From Polymer International (2006), 55(4), 391-398. Language: English, Database: CAPLUS, DOI:10.1002/pi.1907

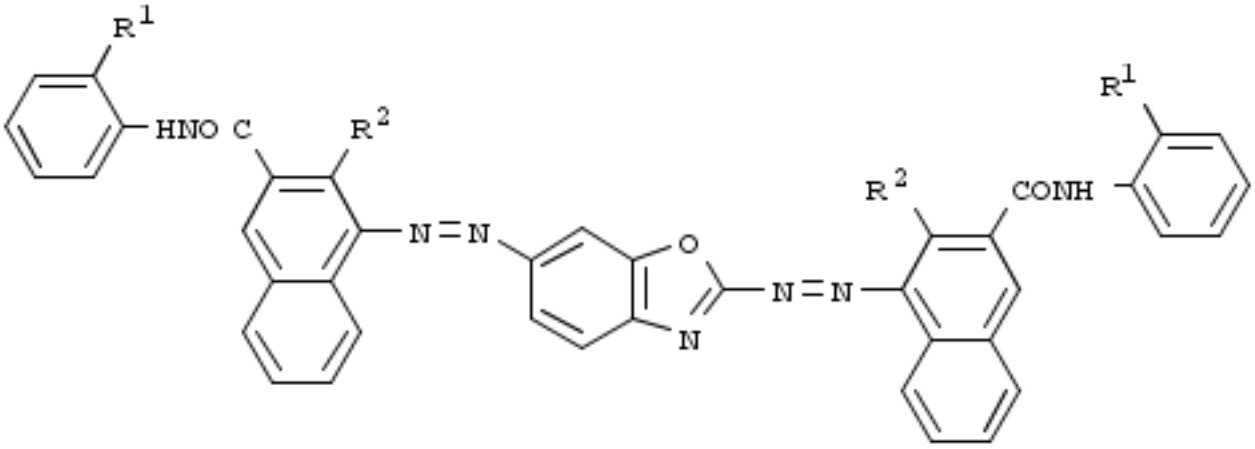
Novel amic acid diamines (AADs) (2-carboxyterephthalamido-bis(alkyl or aryl amine))s, $H_2NXNHOCC_6H_3(COOH)CONHXNH_2$, where X is $(CH_2)_2$, $(CH_2)_3$, $CH_2C_6H_{10}CH_2$, piperazinylethyl, C_6H_4, $C_6H_4OC_6H_4$ or $C_6H_4CH_2C_6H_4$ were synthesized by reacting trimellitic anhydride chloride with arom. or aliph. diamines in DMF at 5°-10°. Poly(amide imide)s (PAIs) with an amide to imide ratio of three in the polymer chains were prepd. by interfacial polycondensation of the AADs in aq. alk. soln. with isophthaloyl chloride or terephthaloyl chloride in dichloromethane at ambient temp. to form poly(amide amic acid)s, followed by their subsequent thermal cycloimidization. All of the PAIs were sol. in polar aprotic solvents such as DMF, dimethylacetamide, DMSO and N-methylpyrrolidone, and have inherent viscosities in the range 0.15-0.48 dL g^{-1}. The polymers were characterized by IR and NMR spectroscopy, TGA and DSC techniques. The PAIs have initial decompn. temps. in the range 250°-460° in air, and glass transition temps. of 128°-320°, depending upon the structures of the monomers. Composite membranes contg. a poly(amide amic acid) and poly(amide imide) barrier layer on the top of a porous polyethersulfone support were prepd. by in situ interfacial polymn. of the AADs in aq. alk. soln. with trimesoyl chloride in hexane, and subsequent curing. The performances of these membranes were evaluated by using aq. feed solns. contg. 2000 ppm NaCl, Na_2SO_4 or $CaCl_2$.

~16 Citings

28. Manufacture of azo/phthalocyanine composite single-layer organic photoconductor

By Yang, Zhenglong; Pu, Hongting
From Faming Zhuanli Shenqing (2005), CN 1632700 A 20050629, Language: Chinese, Database: CAPLUS

The title org. photoconductor with wide frequency response and high photosensitivity is manufd. by mixing carrier generating material, carrier transporting material, resin, and conductive matrix at a certain wt. ratio, and dip-coating to obtain the final product. The carrier generating material is oxazolyl bisazo represented by formula I ($R^{1,2}$ = C_{1-12} alkyl, alkoxy, halogen, hydroxyl, nitro, or carboxyl) / metal phthalocyanine represented by formula II (M = Cu, Co, Ni, Al, etc.) composite material, and the carrier transporting material can be selected from compds. of triaryl amine, hydrazone, pyrazoline, etc. This invention has advantages of simple process, high efficiency, low pollution, etc. The org. photoconductor can be used in xerog. printer with visible light source, and laser printer with light source of near IR laser.



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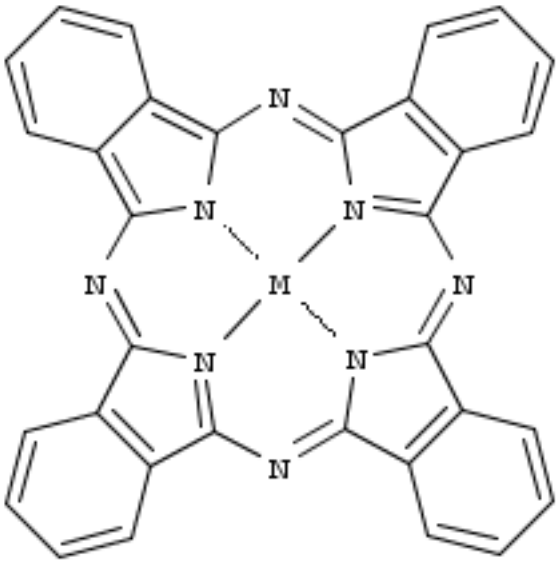


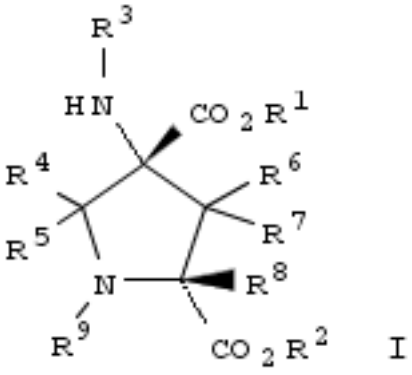
II

~1 Citing

29. Trans pyrrolidinyl derivatives and their pharmaceutical uses

By Schann, Stephan; Acher, Francine
From Eur. Pat. Appl. (2005), EP 1604978 A1 20051214, Language: English, Database: CAPLUS

The present invention relates to the use of trans pyrrolidinyl of the formula I or II (in which: R1, R2 or R3 are H or a carboxy or amino protecting group; R4 to R8 represent H or an alkyl radical; R9 represents a $(R10)_n(-R11)_m$ group wherein n = 0-4, m = 1-3, R10 is -CO-, -CS-, -O-, -S-, -SO-, -SO2-, -COO-, -CON($C_{n'}H_{2n'+1}$)-,-N($C_{n'}H_{2n'+1}$)CO-, -CSN($C_{n'}H_{2n'+1}$)-, -N($C_{n'}H_{2n'+1}$)CS-, -N($C_{n'}H_{2n'+1}$)-, -($C_{n'}H_{2n'+1}$)-, aryl, R11 is a polar group, and n' = 0-8) for the treatment and/or prophylaxis of conditions assocd. with altered glutamatergic signaling and/or functions, and/or conditions which can be affected by alteration of glutamate level or signaling in mammals. General synthetic procedures are described; no biol. data is given.



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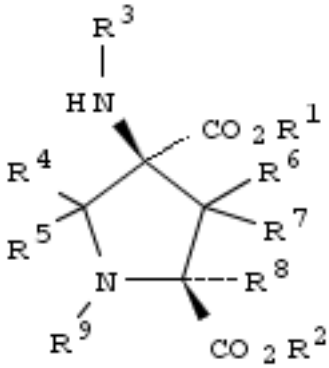


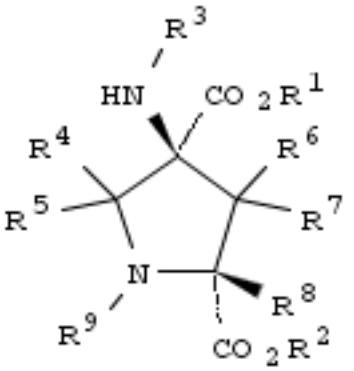
II

~0 Citings

30. Cis-pyrrolidinyl derivatives as modulators of metabotropic glutamate receptors, their preparation, pharmaceutical compositions, and their use in therapy

By Schann, Stephan; Acher, Francine
From Eur. Pat. Appl. (2005), EP 1604977 A1 20051214, Language: English, Database: CAPLUS

The invention relates to cis-pyrrolidinecarboxylic acids of the formula I or II, which are modulators of metabotropic glutamate receptors (mGluRs), either agonists, antagonists, or reverse agonists (no specific data). In compds. I and II, R^1 and R^2 are independently H or a carboxy-protecting group; R^3 is H or an amino-protecting group; R^4 to R^8 are independently selected from H, OH, SH, halo, (un)substituted alkyl, and (un)substituted aryl, or R^4 and R^5 can together form a carbonyl or a thiocarbonyl; and R^9 is selected from carboxy, carboxyalkanoyl, carboxyalkenoyl, carboxyalkyl(thio)carbamoyl, carboxyarylcarbonyl, carboxyarylsulfonyl, hydroxyalkanoyl, alkoxyalkanoyl, aminoalkanoyl, etc.; including pharmaceutically acceptable salts or their metabolically labile esters or amides. The invention also relates to the prepn. of I and II, pharmaceutical compns. comprising a compd. of formula I or II and a pharmaceutically acceptable carrier, as well as to the use of the compns. for the treatment and/or prevention of conditions assocd. with altered glutamatergic signaling and/or functions. A general scheme for the prepn. of the compds. of the invention, e.g., III, is presented, but no specific examples of prepn. are given.



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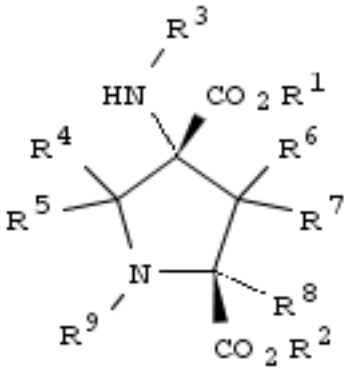

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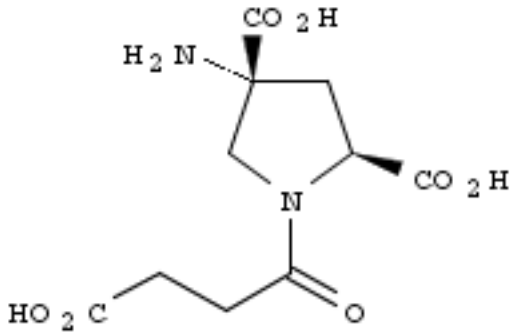


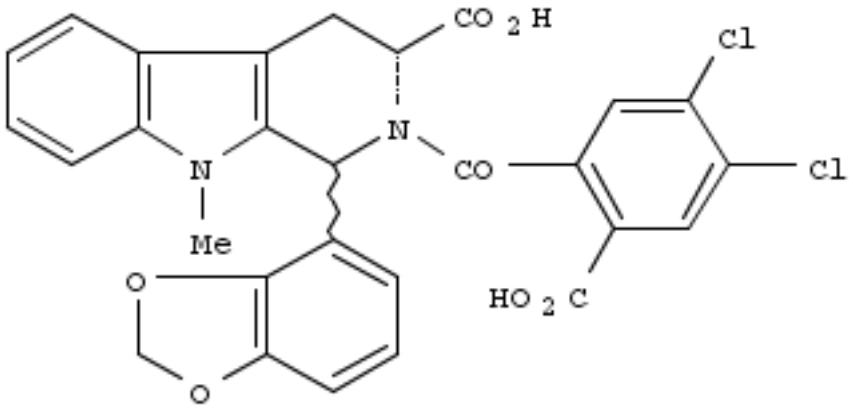
III

~1 Citing

31. Antibiotic tetrahydro-β-carboline derivatives

By Opperman, Timothy; Arvanites, Anthony C.; Pinto, Julia C.; Xiang, Yibin; Ali, Syed Masarrat; Geng, Bolin; Ashwell, Mark A.
From PCT Int. Appl. (2004), WO 2004096802 A2 20041111, Language: English, Database: CAPLUS

Tetrahydro-β-carboline derivs. are disclosed, as are pharmaceutical compns. comprising them and methods using them for treating bacterial infections. The compds. are inhibitors of PPAT (phosphopantetheine adenyltransferase), and are useful in the treatment and prevention of diseases caused by bacteria, particularly bacteria dependent on PPAT, e.g. Escherichia coli, Helicobacter pylori, Staphylococcus aureus, and the like. Prepn. and activity of I are included.



I

~0 Citings

32. Solid state synthesis of some carboxy amides under solvent-free conditions

By Ravinder, V.; Rani, P. Usha; Balaswamy, G.
From Indian Journal of Heterocyclic Chemistry (2004), 14(1), 73-74. Language: English, Database: CAPLUS

Interaction of maleic, succinic and phthalic anhydrides with various amines in solid state furnishes secondary amides with an opening of anhydride rings. One of the resulting amide products 2-mercapto maleanilic acid is undergoing Michael addn. and produces cyclic compd., benz[5,6]-2-carboxymethyl-1,3,4-thiolactam. All the products are obtained in excellent yields and in a state of high purity.

~4 Citings

33. Thalidomide pharmacokinetics and metabolite formation in mice, rabbits, and multiple myeloma patients

By Chung, Francisco; Lu, Jun; Palmer, Brian D.; Kestell, Philip; Browett, Peter; Baguley, Bruce C.; Tingle, Malcolm; Ching, Lai-Ming
From Clinical Cancer Research (2004), 10(17), 5949-5956. Language: English, Database: CAPLUS, DOI:10.1158/1078-0432.CCR-04-0421

Thalidomide has a variety of biol. effects that vary considerably according to the species tested. The authors sought to establish whether differences in pharmacokinetics could form a basis for the species-specific effects of thalidomide. Mice and rabbits were administered thalidomide (2 mg/kg) p.o. or i.v., and plasma concns. of thalidomide were measured after drug administration using high performance liq. chromatog. Plasma samples from five multiple myeloma patients over 24 h after their first dose of thalidomide (200 mg) were similarly analyzed and all data were fitted to a one-compartment model. Metabolites of thalidomide in plasma were identified simultaneously using liq. chromatog.-mass spectrometry. Plasma concn.-time profiles for the individual patients were very similar to each other, but widely different pharmacokinetic properties were found between patients compared with those in mice or rabbits. Area under the concn. curve values for mice, rabbits, and multiple myeloma patients were 4, 8, and 81 μmol/L · hour, resp., and corresponding elimination half-lives were 0.5, 2.2, and 7.3 h, resp. Large differences were also obsd. between the metabolite profiles from the three species. Hydrolysis products were detected for all species, and the proportion of hydroxylated metabolites was higher in mice than in rabbits and undetectable in patients. Our results show major interspecies differences in the pharmacokinetics of thalidomide that are related to the altered degree of metab. The authors suggest that the interspecies differences in biol. effects of thalidomide may be attributable, at least in part, to the differences in its metab. and hence pharmacokinetics.

~30 Citings

34. Metabolism of thalidomide in liver microsomes of mice, rabbits, and humans

By Lu, Jun; Helsby, Nuala; Palmer, Brian D.; Tingle, Malcolm; Baguley, Bruce C.; Kestell, Philip; Ching, Lai-Ming
From Journal of Pharmacology and Experimental Therapeutics (2004), 310(2), 571-577. Language: English, Database: CAPLUS, DOI:10.1124/jpet.104.067793

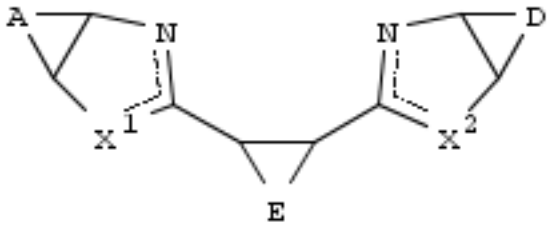
Thalidomide is increasingly important in clin. treatment, not only of various inflammatory conditions but also in multiple myeloma and other malignancies. Moreover, the metab. of thalidomide varies considerably among different species, indicating a need to understand its mechanistic basis. Our previous in vivo studies showed the plasma half-life of thalidomide to be much shorter in mice than in humans, with rabbits showing intermediate values. The authors were unable to detect hydroxylated thalidomide metabolites in humans and suggested that interspecies differences in thalidomide hydroxylation might account for the differences in plasma half-life. The authors sought here to establish whether these species differences in the formation of hydroxylated thalidomide metabolites could be discerned from in vitro studies. Liver microsomes of mice, rabbit, and human donors were incubated with thalidomide and analyzed using liq. chromatog.-mass spectrometry. Hydrolysis products were detected for all three species, and the rates of formation were similar to those for spontaneous hydrolysis, except in rabbits where phthaloylisoglutamine formation increased linearly with microsomal enzyme concn. Multiple hydroxylation products were detected, including three dihydroxylated metabolites not obsd. in vivo. Thalidomide-5-O-glucuronide, detected in vivo, was absent in vitro. The amt. of 5-hydroxythalidomide formed was high in mice, lower in rabbits, and barely detectable in humans. The authors conclude that major interspecies differences in hepatic metab. of thalidomide relate closely to the rate of in vivo metabolite formation. The very low rate of in vitro and in vivo hydroxylation in humans strongly suggests that thalidomide hydroxylation is not a requirement for clin. anticancer activity.

~33 Citings

35. Preparation of bis-benzimidazoles and related compounds as potassium channel modulators

By Wang, Xiaodong; Fulp, Alan Bradley; Van Rhee, Albert Michiel; Spear, Kerry Leigh
From PCT Int. Appl. (2003), WO 2003094861 A2 20031120, Language: English, Database: CAPLUS

Title compds. I [wherein A, D, and E = independently (un)substituted (hetero)aryl; X^1 and X^2 = independently NR^1, S, O, NCH_2R^2, CR^3, or CHR^4; R^1-R^4 = independently H or (un)substituted alkyl, or heteroaryl] were prepd. as modulators of Ca^{2+}-activated K^+ (SK) channels. For example, 5-nitroisobenzofuran-1,3-dione was coupled with 1,2-phenylenediamine to give 5-nitro-2-[2-(5-nitro-1H-benzimidazol-2-yl)phenyl]benzimidazole, which was reduced the the diamine II. Six representative compds. of the invention were assayed and exhibited activity toward hSK channels. Thus, I and their pharmaceutical compns. are of use in both therapeutic and diagnostic methods (no data).



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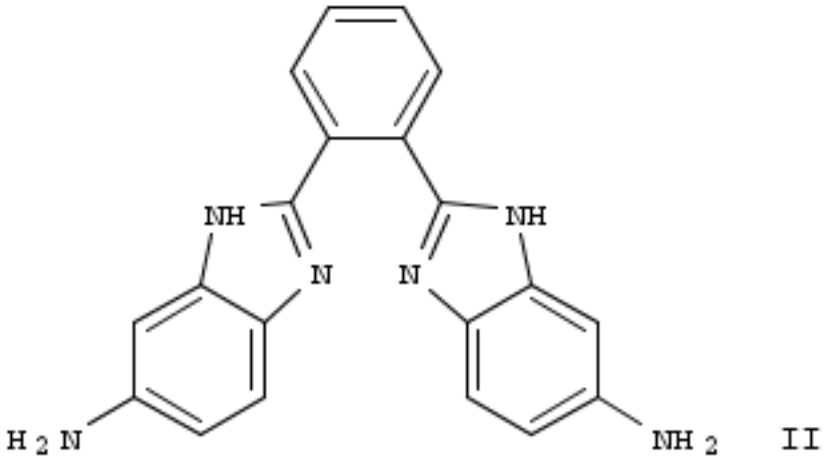


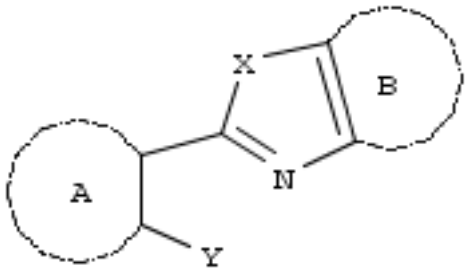
II

~8 Citings

36. Preparation of phenylbenzimidazoles as osteoclast differentiation induction inhibitors and osteoclast inhibitors

By Nakahira, Hiroyuki; Horiuchi, Yoshihiro
From Jpn. Kokai Tokkyo Koho (2002), JP 2002161084 A 20020604, Language: Japanese, Database: CAPLUS

The compds. I [ring A, ring B = (un)substituted arom. ring; X = NR^0, S, O; R^0 = H, lower alkyl; Y = NR^1R^2, $CONR^{1'}R^{2'}$, $C(OH)R^{1''}R^{2''}$, (un)substituted (un)satd. 5- to 7-membered heterocycle; R^1 = (un)substituted lower alkyl, alkenyl, alkynyl; R^2 = org. group excluding lower alkyl; R^1R^2 may form heterocycle; $R^{1'}$, $R^{2'}$ = (un)substituted lower alkyl; $R^{1'}R^{2'}$ may form heterocycle; $R^{1''}$, $R^{2''}$ = (un)substituted lower alkyl] or their pharmaceutically acceptable salts are prepd. The compds. are useful for anti-inflammatory agents, antirheumatic agents, and agents for bone regeneration. 2-(5,6-Dichloro-1H-imidazol-2-yl)-N-methylaniline (2.06 g) was reacted with acetyl chloride in pyridine at 25° for 1 h to give 630 mg N-[2-(5,6-dichloro-1H-benzimidazol-2-yl)phenyl]-N-methylacetamide showing 66% inhibition of osteoclast differentiation in vitro.



I

~8 Citings

37. Studies on iridium(III) complexes with ligands containing amide group

By Dayakar, G.; Lingaiah, P.
From Asian Journal of Chemistry (2001), 13(3), 1105-1108. Language: English, Database: CAPLUS

Complexes of Ir(III) with 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, 2-(aminobenzoylamino)benzoic acid, 2-[(2-aminophenylamino)carbonyl]benzoic acid, maleanilic acid, malea-1-naphthalanilic acid, 2-[(phenylamino)carbonyl]benzoic acid were synthesized and characterized by physicochem. data.

~1 Citing

38. Structure and reactions of monoanils obtained from 2,3-pyridinediamines

By Dubey, P. K.; Kulkarni, Subhash M.; Kumar, R. Vinod
From Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry (2001), 40B(5), 361-367. Language: English, Database: CAPLUS

The reaction of 2,3-pyridinediamines with arom. aldehydes results in the formation of 2-amino-3-arylideneaminopyridines (I), resp. Dehydrogenative cyclization of I with different reagents give 2-aryl-1H-imidazo[4,5-b]pyridines. Reactions of I with different reagents have been described.

~5 Citings

39. Synthesis and cytotoxic activity of carboxamide derivatives of benzimidazo[2,1-a]isoquinoline and pyrido[3',2':4,5]imidazo[2,1-a]isoquinoline

By Deady, Leslie W.; Rodemann, Thomas; Finlay, Graeme J.; Baguley, Bruce C.; Denny, William A.
From Anti-Cancer Drug Design (2001), 15(5), 339-346. Language: English, Database: CAPLUS

A series of benzimidazo[2,1-a]isoquinolines with carboxamide side chains at the 1-, 6-, 9- and 11-positions were prepd., to study the biol. effects of varying the position of the side chain in this tetracyclic series. The 6-, 9- and 11-analogs were obtained by modifications to published chem. The 1-carboxamide analog was obtained via a one-pot isocoumarin/isoquinolone conversion of 3-methylisocoumarin-8-carboxylic acid with o-phenylenediamine in buffered aq. acid, which gave the required 1-acid. The compds. were evaluated in a panel of cell lines in culture. The 6-carboxamides, where the side chain is attached to one of the central rings, were not active, but the 1- and 11-carboxamides, where the side chain is attached to one of the terminal rings off the chromophore short axis, were reasonably cytotoxic (IC_{50}s < 1 μM). Overall, the structure-activity relationships are broadly in line with those seen with other tri- and tetracyclic carboxamides, and are consistent with recent crystal structure studies of acridine-4-carboxamides bound to DNA. The most potent 1-carboxamide was highly active in vivo against s.c. colon 38 tumors in mice, providing a growth delay of 12 days.

~21 Citings

40. Polyimides starting from bis(o-amino)phenols or aromatic tetraamines: synthesis and transformation chemistry

By Likhatchev, D.; Granados-Focil, S.; Guzman-Lucero, D.; Ruiz-Rojas, B. L.
From Annual Technical Conference - Society of Plastics Engineers (2000), 58th(Vol. 3), 3599-3603. Language: Spanish, Database: CAPLUS

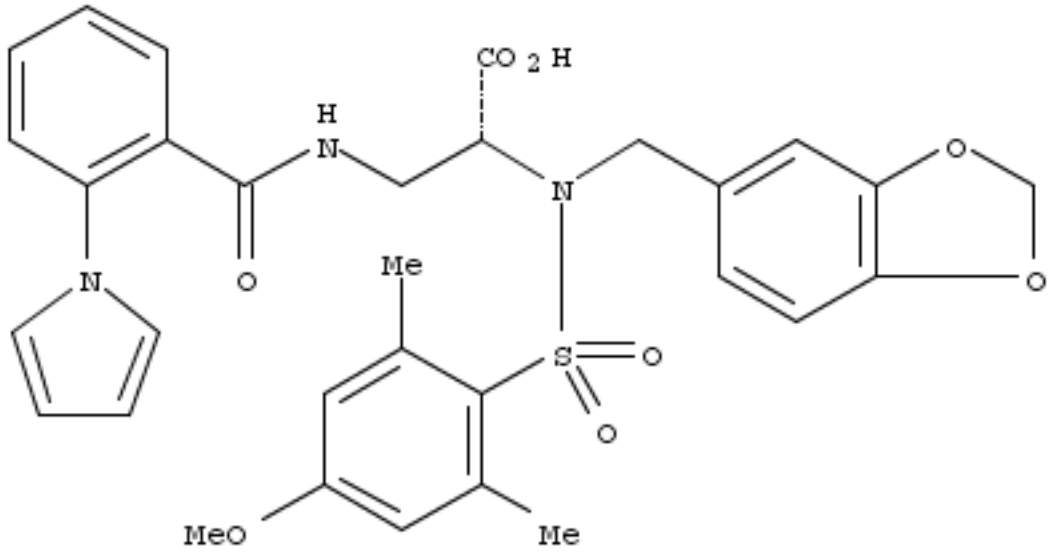
Several ortho-substituted poly(pyromellitimide)s were prepd. by treatment of pyromellitic dianhydride-3,3'-diaminobenzidine copolymer with acetic anhydride or trifluoroacetic anhydride or by thermal treatment (300°, 30 min). The cyclodehydration of precursors contg. OH or NH_2 groups at the ortho position to N in diamine fragments in the presence of aliph. anhydrides and tertiary amines took place with formation of o-acetate and o-acetamide side groups, resp. The model compds., e.g., 2-(o-carboxyphenyl)benzimidazole, and 2'-Amino-N-phenylphthalimide underwent spontaneous cyclodehydration to form the corresponding imide at ambient temp. in aq. medium and without dehydration agents. Treatment of N-(2-aminophenyl)phthalamic acid con trifluoroacetic anhydride led to formation of the isoimide with a trifluoroacetamide group at the ortho position of the amine N. This compd. is thermodynamically unstable and underwent a second cyclodehydration to form a ladder structure of 1,2-benzoylenbenzimidazol. The yield of this reaction increased with heterocycle size.

~0 Citings

41. Preparation of amino acid sulfonamide hydroxamates as inhibitors of procollagen C-proteinase.

By Billedeau, Roland Joseph; Broka, Chris Allen; Campbell, Jeffrey Allen; Chen, Jian Jeffrey; Dankwardt, Sharon Marie; Delaet, Nancy; Robinson, Leslie Ann; Walker, Keith Adrian Murray
From PCT Int. Appl. (2000), WO 2000037436 A1 20000629, Language: English, Database: CAPLUS

$HOHNCOCHR^1NRSO_2Ar^2$ [R^1 = alkyl, haloalkyl, heteroalkyl, cycloalkyl, aryl, aralkyl, aralkenyl, heteroaryl, heteroaralkyl, aminl, aryl, aralkyl, etc.; R = CHR^2Ar^1, $CHR^2CH{:}CHAr^1$; Ar^2 = specified (substituted) Ph, naphthyl; R^2 = H, alkyl; with provisos], were prepd. Thus, N-hydroxy-2(R)-[(3,4-methylenedioxybenzyl)(4-methoxy-2,3,6-trimethylbenzenesulfonyl)amino]-3-methylbutyramide was prepd. by soln. phase synthesis from BOC-D-Val-OH. Title compds. inhibited procollagen C-proteinase with IC_{50} 0.01-2 μM.



~11 Citings

42. Synthesis and characterization of Poly(amide-imide)s and their precursors as materials for membranes

By Reddy, A. V. Rami
From Journal of Applied Polymer Science (2000), 75(14), 1721-1727. Language: English, Database: CAPLUS

Water sol. diamine amic acids (DAAs) were synthesized by reacting aliph. diamines with pyromellitic dianhydride. Poly(amide-amic acids) (PAAs) were prepd. by interfacial polycondensation of DAAs in aq. sodium hydroxide soln. with isophthaloyl chloride in dichloromethane. Poly(amide-imides) (PAIs) contg. alternating (amide-amide)-(imide-imide) sequences were obtained by thermal cycloimidization of the PAA films at 175°C for 4 h in a forced air woven. The PAIs were readily sol. in polar aprotic solvents such as DMF, dimethylacetamide, dimethylsulfoxide, and N-methyl-2-pyrrolidone. The inherent viscosities of the polymers are in the range of 0.97-1.7 dL/g. The polymers were characterized by IR, ^{1}H NMR, and thermogravimetric anal. Thin film composite membranes contg. PAA ultrathin barrier layer were prepd. by in situ interfacial polycondensation of DAA in water with trimesoyl chloride or isophthaloyl chloride in hexane on the surface of a porous polysulfone membrane. The membranes were characterized for water permeability and for the sepn. of NaCl and Na_2SO_4.

~2 Citings

43. Low-temperature route to 1,2-benzoylenebenzimidazole ladder structure

By Likhatchev, D.; Granados-Focil, S.; Gavino, R.; Canseco, M.; Alexandrova, L.
From High Performance Polymers (1999), 11(4), 405-415. Language: English, Database: CAPLUS, DOI:10.1088/0954-0083/11/4/306

Low-temp. catalytic conversion of N-(o-aminophenyl)phthalamic acid (I) was studied as a model reaction for the synthesis of ladder poly(aroylenebenzimidazoles). The treatment of I with acetic anhydride/pyridine or trifluoroacetic anhydride resulted in the selective and quant. cyclodehydration to yield either imide or isoimide structures, resp. The imidization was accompanied by acylation of the ortho amino group to form lateral acetamide or trifluoroacetamide substituents. Thermodynamically unstable N-[o-(trifluoroacetamido)phenyl]phthalisoimide (II) underwent secondary cyclization to yield ladder 1,2-benzoylenebenzimidazole (III) between 130 and 150°C. The conversion of this reaction did not exceed 35% because it competed with the thermal isomerization of II to the stable N-[o-(trifluoroacetamido)phenyl]phthalimide. The cyclization of II was found to be possible even at room temp. The formation of 30-35% of III was obsd. after II was dissolved in DMF and stored at room temp. for 4-10 h. This also was accompanied by isoimide-to-imide isomerization. The obtained data may be useful for the further development of novel low-temp. approaches to the synthesis of ladder poly(aroylenebenzimidazoles). Synthesis of arom. polyimides and polyisoimides with lateral alkylamide or trifluoroalkylamide from available arom. dianhydrides and tetraamines may also be of practical interest.

~4 Citings

44. Theoretical study of N-(o-aminophenyl amic) acid cyclodehydration to 1,2-benzoylenebenzimidazole as a model reaction of ladder polypyrrones synthesis: thermodynamic and thermochemical data

By Salcedo, Roberto; Valle, Leticia; Alexandrova, Larissa; Likhatchev, Dmitri
From Journal of Molecular Structure: THEOCHEM (1999), 463(3), 231-235. Language: English, Database: CAPLUS, DOI:10.1016/S0166-1280(98)00132-8

The mechanism of polyimidazopyrrolones formation was studied via thermodn. simulation of N-(o-aminophenyl)amic acid cyclodehydration to 1,2-benzoylenebenzimidazole. It was found that 2-(o-carboxyphenyl)benzimidazole and N-(o-aminophenyl)phthalimide were the most thermodynamically favorable intermediates of this process. The thermodn. possibility of 1,2-benzoylenebenzimidazole formation from N-(o-acetamidophenyl)phthalimide and N-(o-trifluoroacetamidophenyl)isophthalimide was also evaluated using the same method. All thermodn. values were obtained from semiempirical quantum mechanics calcns.

~2 Citings

45. Synthesis and spectral studies of dioxouranium(VI) complexes of amide group containing ligands

By Dayakar, G.; Lingaiah, P.
From Asian Journal of Chemistry (1999), 11(1), 137-140. Language: English, Database: CAPLUS

Complexes of dioxouranium(VI) with 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, 2-(2-aminobenzoylamino)benzoic acid, 2-aminobenzanilide, 2-(aminocarbonyl)benzoic acid, 2-[(2-aminophenylamino)carbonyl]benzoic acid, maleanilic acid, maleo-1-naphthanilic acid, 2-[(phenylamino)carbonyl]benzoic acid and 2-[(2-naphthalenylamino)carbonyl]benzoic acid were synthesized and characterized by physicochem. data.

~1 Citing

46. Chiral Inversion and Hydrolysis of Thalidomide: Mechanisms and Catalysis by Bases and Serum Albumin, and Chiral Stability of Teratogenic Metabolites

By Reist, Marianne; Carrupt, Pierre-Alain; Francotte, Eric; Testa, Bernard
From Chemical Research in Toxicology (1998), 11(12), 1521-1528. Language: English, Database: CAPLUS, DOI:10.1021/TX9801817

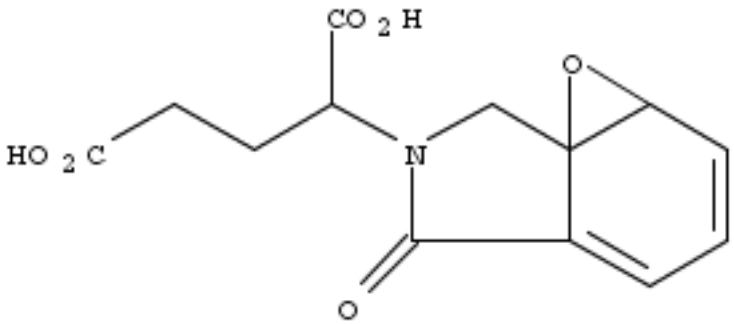
The chiral inversion and hydrolysis of thalidomide and the catalysis by bases and human serum albumin were investigated by using a stereoselective HPLC assay. Chiral inversion was catalyzed by albumin, hydroxyl ions, phosphate, and amino acids. Basic amino acids (Arg and Lys) had a superior potency in catalyzing chiral inversion compared to acid and neutral ones. The chiral inversion of thalidomide is thus subject to specific and general base catalysis, and it is suggested that the ability of HSA to catalyze the reaction is due to the basic groups of the amino acids Arg and Lys and not to a single catalytic site on the macromol. The hydrolysis of thalidomide was also base-catalyzed. However, albumin had no effect on hydrolysis, and there was no difference between the catalytic potencies of acidic, neutral, and basic amino acids. This may be explained by different reaction mechanisms of the chiral inversion and hydrolysis of thalidomide. Chiral inversion is deduced to occur by electrophilic substitution involving specific and general base catalysis, whereas hydrolysis is thought to occur by nucleophilic substitution involving specific and general base as well as nucleophilic catalysis. As nucleophilic attack is sensitive to steric properties of the catalyst, steric hindrance might be the reason albumin is not able to catalyze hydrolysis. ^{1}H NMR expts. revealed that the three teratogenic metabolites of thalidomide, in sharp contrast to the drug itself, had complete chiral stability. This leads to the speculation that, were some enantioselectivity to exist in the teratogenicity of thalidomide, it could result from fast hydrolysis to chirally stable teratogenic metabolites.

~89 Citings

47. Methods and compositions using thalidomide or other angiogenesis-inhibitory compound and anti-inflammatory agent for inhibition of angiogenesis

By D'Amato, Robert J.
From PCT Int. Appl. (1998), WO 9819649 A2 19980514, Language: English, Database: CAPLUS

A group of compds. that effectively inhibit angiogenesis is provided. More specifically, thalidomide and various related compds.,e.g. thalidomide precursors, analogs, metabolites and hydrolysis products, have been shown to inhibit angiogenesis and to treat disease states resulting from angiogenesis. Addnl., antiinflammatory drugs, such as steroids and NSAIDs can inhibit angiogenesis-dependent diseases either alone or in combination with thalidomide and related compds. Importantly, these compds. can be administered orally.



~19 Citings

48. Spontaneous cyclodehydration of N- (o-aminophenyl) amic acids

By Likhatchev, D.; Valle, L.; Canseco, M.; Salcedo, R.; Gavino, R.; Martinez-Richa, A.; Alexandrova, L.; Vera-Graziano, R.
From Journal of Applied Polymer Science (1998), 67(4), 609-619. Language: English, Database: CAPLUS, DOI:10.1002/(SICI)1097-4628(19980124)67:4<609::AID-APP3>3.0.CO;2-C

The model reaction between phthalic anhydride and 1,2-diaminobenzene was studied under conditions analogous to those of the low-temp. polycondensation of arom. dianhydrides with bis(o-diamines) in amide solvents followed by thermal cyclodehydration in condensed state to form ladder poly(imidazo pyrrolones) (poly-pyrrones). The intermediate N-(o-aminophenyl)phthalamic acid undergoes spontaneous cyclodehydration to give N-(o-aminophenyl)phthalimide and 2-(o-carboxy-phenyl)benzimidazole. The reaction occurred at ambient temp. in the presence of water or alc. without using a dehydration agent. The yield of imide-amine and/or carboxy-benzimidazole depended on the temp. of the condensation reaction. Temps. below 0° appeared to favor the formation of the carboxy-benzimidazole. Thermal cyclization of N-(o-aminophenyl)phthalamic acid occurred through the same intermediates: imide-amine and carboxy-benzimidazole. The former converted to the pyrrone above 200°, while the secondary cyclization of the latter started above 250°. Spontaneous cyclodehydration was also obsd. for polyamide acid-amine precursors obtained by low-temp. polycondensation of pyromellitic dianhydride with 3,3'-diaminobenzidine. The prepolymer solns. in DMF dild. with water at room temp. turned to a gel after 48-72 h. A spectroscopic study of the resulting polymers indicated the presence of significant amts. of cyclic imides.

~7 Citings

49. A new kinetic model for the acid-catalyzed reactions of N-(2-aminophenyl)phthalamic acid in aqueous media

By Perry, Christopher J.
From Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1997), (5), 977-982.
Language: English, Database: CAPLUS

The acid-catalyzed breakdown of N-(2-aminophenyl)phthalamic acid has been studied in dil. aq. acids in the pH range 0-6. The dominant reaction is the formation of N-(2-aminophenyl)phthalimide (between ~80 and ~100% yields in the pH range studied) and its subsequent rearrangement to 2-(2-carboxyphenyl)benzimidazole, occurring as consecutive pseudo-first-order processes. Anomalously, only a minor hydrolysis reaction is obsd. A kinetic model for these processes has been constructed and rate consts. and activation parameters evaluated. Mechanisms involving pre-equil. to form the kinetically significant species have been proposed for the consecutive processes. The approach has been adapted to account for the obsd. kinetics of acid catalyzed formation of benzimidazoles from o-aminoanilides.

~12 Citings

50. Synthesis and spectral studies of ruthenium(III) complexes with amide group ligands

By Dayakar, G.; Lingaiah, P.
From Asian Journal of Chemistry (1997), 9(2), 179-182. Language: English, Database: CAPLUS

Ru(III) Complexes of a few amide group ligands such as 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, 2-(2-aminobenzoylamino)benzoic acid, 2-(aminobenzanilide), 2-(aminocarbonyl)benzoic acid, 2-[(2-aminophenylamino)carbonyl]benzoic acid, maleanilic acid, malea-1-naphthalanilic acid, 2-[(phenylamino)carbonyl]benzoic acid and 2-[(2-naphthalenylamino)carbonyl]benzoic acid were synthesized and characterized by physicochem. data.

~5 Citings

51. Methods and compositions for inhibition of angiogenesis

By D'Amato, Robert
From U.S. (1997), US 5593990 A 19970114, Language: English, Database: CAPLUS

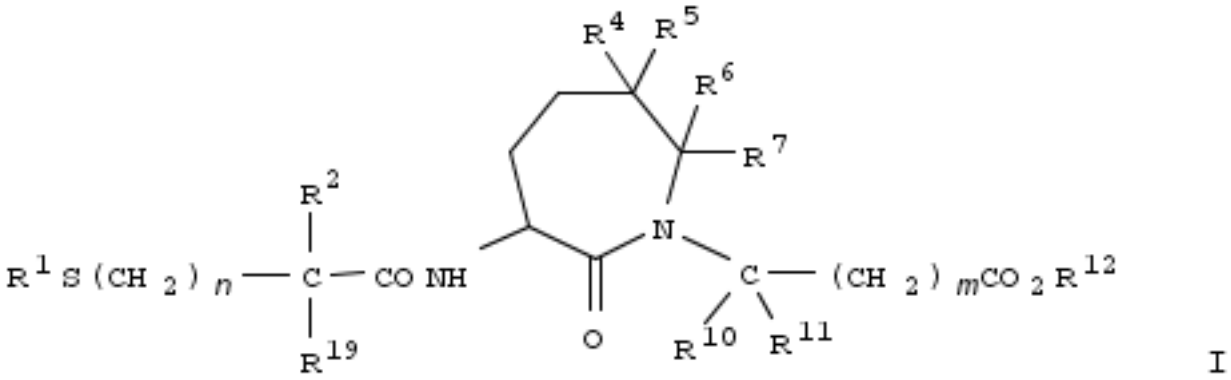
The present invention comprises a group of compds. that effectively inhibit angiogenesis. More specifically, thalidomide and various related compds. such as thalidomide precursors, analogs, metabolites and hydrolysis products have been shown to inhibit angiogenesis and to treat disease states resulting from angiogenesis. Importantly, these compds. can be administered orally.

~19 Citings

52. Substituted azepinone dual inhibitors of angiotensin-converting enzyme and neutral endopeptidase

By Karanewsky, Donald S.; Robl, Jeffrey A.
From U.S. (1996), US 5552397 A 19960903, Language: English, Database: CAPLUS

Azepinones I [R^1 = H, acyl, (substituted) alkylthio; R^2, R^{19} = H, (substituted) alkyl; R^4, R^5 = H, (substituted) alk(en)yl; or R^4 = H, R^5 = OH; or R^4R^5 = :O; R^6, R^7, R^{10}, R^{11} = H, (substituted) alk(en)yl; R^{12} = (substituted) alkyl, (substituted) acyloxymethyl; m, n = 0, 1] are disclosed which possess inhibitory activity against angiotensin-converting enzyme and neutral endopeptidase and thus are useful as cardiovascular agents. Thus, (S)-2-(acetylthio)benzenepropanoic acid was amidated with Et (S)-2,3,4,5-tetrahydro-3-amino-2-oxo-1H-benzazepine-1-acetate and sapond. to produce [S-(R*,R*)]-2,3,4,5-tetrahydro-3-[(2-mercapto-1-oxo-3-phenylpropyl)amino]-2-oxo-1H-benzazepine-1-acetic acid (II). Tablets were prepd. each contg. the 2-acetic acid analog of II 200, corn starch 100, gelatin 20, Avicel 50, and Mg stearate 5 mg.



I

~21 Citings

53. Studies on rhodium(III) complexes with ligands containing amide group

By Dayakar, G.; Lingaiah, P.
From Indian Journal of Chemistry, Section A: Inorganic, Bio-inorganic, Physical, Theoretical & Analytical Chemistry (1996), 35A(7), 614-616. Language: English, Database: CAPLUS

Complexes of Rh(III) with 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, 2-(2-aminobenzoylamino)benzoic acid, 2-(aminobenzanilide), 2-(aminocarbonyl)benzoic acid, 2-[(2-aminophenylamino)carbonyl]benzoic acid, maleanilic acid, malea-1-naphthalanilic acid, and 2-[(phenylamino)carbonyl]benzoic acid were prepd. and characterized by elemental anal., conductance, thermal, magnetic, IR, NMR and electronic spectral data. The complexes are octahedral.

~5 Citings

54. Interaction of 3-N-(w-carboxyacyl)-L-2,3-diaminopropionic acids with hippocampal synaptic membranes

By Koreshonkov, O. N.; Perestenko, P. V.; Dumpis, M. A.; Piotrovskii, L. B.
From Khimiko-Farmatsevticheskii Zhurnal (1995), 29(8), 3-6. Language: Russian, Database: CAPLUS

A new analog of 3-N-(w-carboxyacyl)-L-2,3-diaminopropionic acid was synthesized and the inhibitory activity of five 3-N-(w-carboxyacyl)-L-2,3-diaminopropionic acid analogs on L-glutamic acid interaction with hippocampal synaptic membranes of humans was studied.

~0 Citings

55. Synthesis and some pharmacological properties of ODAP analogs

By Piotrovsky, L. B.; Dumpis, M. A.; Garyaev, A. P.; Zaitzev, Yu. V.
Edited By:Sarel, Shalom; Mechoulam, Raphael; Agranat, Israel
From Trends Med. Chem. '90, Proc. Int. Symp. Med. Chem., 11th (1992), 183-9. Language: English, Database: CAPLUS

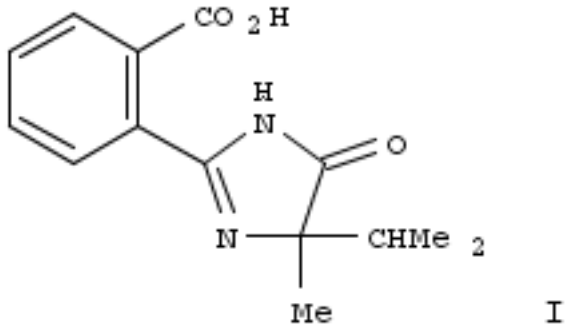
A symposium report on the prepn. and pharmacol. activity of N^3-oxalyl-L-2,3-diaminopropionic acid (ODAP) and its N^3-succinyl- (SuDAP), N^3-glutaryl- (GIDAP) and N^3-phthalamoyl- (PtDAP) analogs in mice after i.c.v. and i.p. administrations. After i.c.v. injection ODAP and SuDAP induced the generalized clonic/tonic seizures, GIDAP and PtDAP could block the convulsive action of NMDA, but not kainate and ODAP. The influences of GIDAP and PtDAP on spontaneous locomotor activity and their antinociceptive action were studied. In contrast to ODAP these compds. possessed the general depressive properties. The increase of the distances between the pharmacophoric groups in ODAP-like mols. convert the quisqualate-preferring agonist to NMDA-preferring antagonists.

~1 Citing

56. Synthesis of imidazolinone herbicide - Ke Cao Nin

By Sun, Xiaohong; Hua, Chengwen; Li, Wenhong; Chen, Bang; Liu, Yuanfa
From Xibei Daxue Xuebao, Ziran Kexueban (1994), 24(3), 227-30, 234. Language: Chinese, Database: CAPLUS

The title compd. (I) was prepd. in 3 steps in 74% overall yield by condensation of phthalic anhydride with 2-amino-2,3-dimethylbutyronitrile followed by hydrolysis and cyclization.



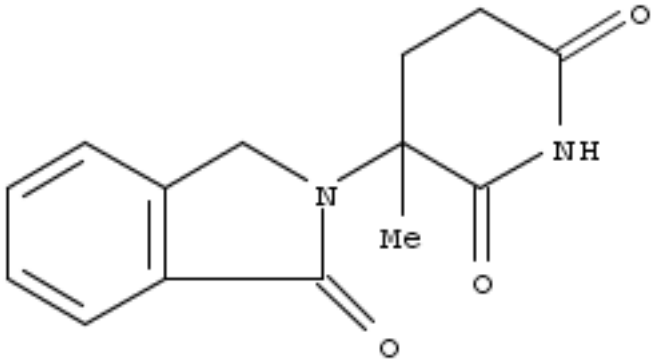
I

~1 Citing

57. Thalidomide compounds in methods and compositions for inhibition of angiogenesis

By D. Amato, Robert
From PCT Int. Appl. (1994), WO 9420085 A1 19940915, Language: English, Database: CAPLUS

The present invention comprises a group of compds. that effectively inhibit angiogenesis. More specifically, thalidomide and various related compds. such as thalidomide precursors, analogs, metabolites and hydrolysis products have been shown to inhibit angiogenesis. Importantly, these compds. can be administered orally. EM-12 was tested in the rabbit cornea angiogenesis assay at 100 and 200mg/kg/day and showed 21% and 43% inhibition, resp.



~15 Citings

58. Study of cyclodehydration and hydrolysis of ortho-aminoanilides. VI. Kinetics and mechanism of isomerization and hydrolysis of N-(2-aminophenyl)phthalimide in solutions of H2SO4

By Shchel'tsin, V. K.; Sycheva, E. A.; Vinnik, M. I.
From Kinetika i Kataliz (1994), 35(2), 249-54. Language: Russian, Database: CAPLUS

In aq. H_2SO_4 soln., 2-$HO_2CC_6H_4CONHC_6H_4NH_2$-2 (1) and N-(2-aminophenyl)phthalimide (3) isomerize to 2-(2-carboxyphenyl)benzimidazole (2), and hydrolyze to o-phenylenediamine and phthalic acid/anhydride. Hydrolysis of 3 and its isomerization to 2 proceeds with significant rate at temp. > 40°; hydrolysis occurs throughout the H_2SO_4 concn. range, and isomerization - at H_2SO_4 concn. < 55%. In 0.05-5% H_2SO_4 the rate of isomerization of 3 increases with degree of ionization of the amino group; the decrease in isomerization at higher concns. of H_2SO_4 was attributed to decrease in concn. of its reactive form (3.H^+) by, e.g., ion pairing. The rate of hydrolysis of 3 increases with H_2SO_4 concn. to 50-60%, and then decreases.

~0 Citings

59. Novel N-substituted imidazolinones. Synthesis and herbicidal activity

By Guaciaro, M. A.; Los, M.; Litle, D. L.; Marc, P. A.; Quakenbush, L.
From ACS Symposium Series (1992), 504(Synth. Chem. Agrochem. III), 56-74. Language: English, Database: CAPLUS, DOI:10.1021/bk-1992-0504.ch007

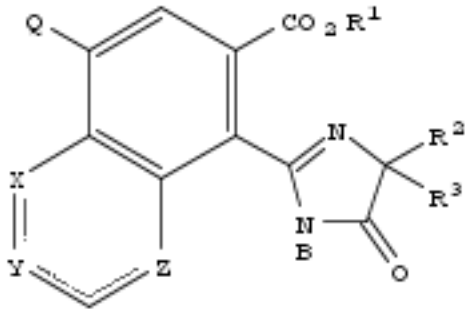
In an effort to prep. new imidazolinone herbicides with the required combination of weed control, crop safety and reduced soil persistence, various novel N-hydroxy, N-chloro- and N-cyanoimidazolinones were synthesized. The N-hydroxyimidazolinones, although interesting from a synthetic standpoint, did not show a significant advantage over their imidazolinone counterparts in the greenhouse. The N-chloroimidazolinones showed better weed control and crop safety than their imidazolinone precursors in several instances but showed no evidence of reduced soil persistence. The N-cyanoimidazolinones exhibited excellent weed control and crop selectivity with evidence of reduced soil persistence, depending upon the nature of the carboxylate substituent.

~1 Citing

60. Preparation of herbicidal 2-(imidazolin-2-yl)-benzo-(6-membered)-heterocycles

By Doehner, Robert Francis, Jr.
From S. African (1992), ZA 9102722 A 19920129, Language: English, Database: CAPLUS

The title compds. [I-IV; R^1 = H, di(C_{1-4} alkyl)amino, (un)substituted C_{1-12} alkyl, -C_{3-12} alkenyl, -C_{3-6} cycloalkyl, -C_{3-16} alkynyl, a cation; R^2 = C_{1-4} alkyl; R^3 = C_{1-4} alkyl, C_{3-6} cycloalkyl, etc.; B = H, COR^4, SO_2R^5; R^4 = C_{1-11} alkyl, $ClCH_2$, (un)substituted Ph; X, Y, Z = CR^6, CR^7R^8, N, NR^9, O, S; dashed line = optional double bond; R^6-R^8 = H, halo, C_{1-4} alkoxy, etc.; R^9 = H, (un)substituted C_{1-4} alkyl, etc.; Q = H, halo, (un)substituted C_{1-4} alkyl, etc.; when $R^{10}R^{11}$ = O then R^{12} = R^2 and R^{13} = R^3; when $R^{12}R^{13}$ = O then R^{10} = R^2 and R^{11} = R^3; provisos are given] were prepd. A mixt. of pyrroloquinolineacetamide (V) [2-step prepn. from benzo[h]quinolin-10-ol given] and 5N NaOH was stirred for 4 h at 100°, cooled to room temp., acidified to pH 4, and extd. to give a brown gum. This was heated with Ac_2O and pyridine for 5 min on a steam bath, evapd., the residue in MeOH treated by NaOMe powder to pH 10-12, stirred overnight, and neutralized by AcOH to give a mixt. of isomeric imidazolinylquinolinecarboxylates which were sepd. by flash chromatog. on silica gel. One of the isomers: Me 8-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-7-quinolinecarboxylate was sapond. by 2N NaOH and the mixt. acidified to give the title compd. (I; B = R^1 = H, R^2 = Me, R^3 = $CHMe_2$, Z = N) (VI; XY = CH:CH). The latter at 0.250 kg/ha preemergence gave complete kill of Convolvulus arvensis and almost complete kill of Cyperus rotundus with a trace effect on wheat and soybean. A quinoxalinecarboxylate analog (VI; XY = N:CH) caused injury on Agropyron repens and Brassica kaber at 0.500 kg/ha.



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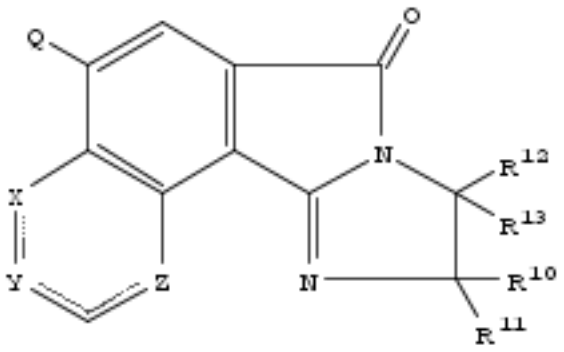

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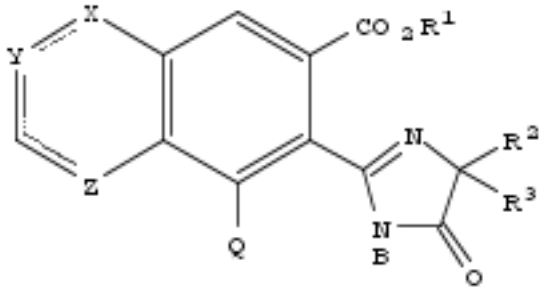

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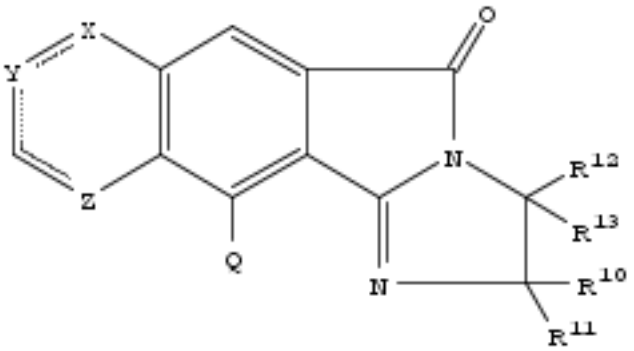

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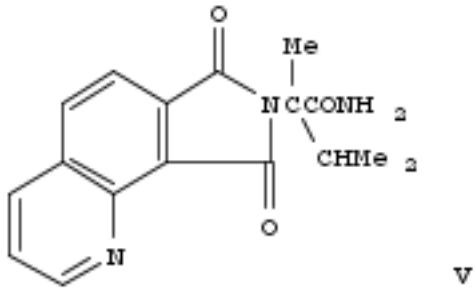


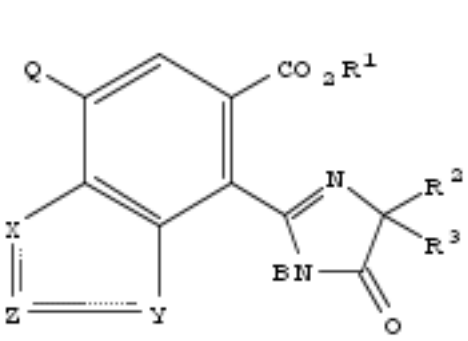
V

~0 Citings

61. Preparation of 2-imidazolonyl heteroannelated benzoates and analogs as herbicides

By Gange, David Michael; Guaciaro, Michael Anthony; Doehner, Robert Francis, Jr.
From S. African (1992), ZA 9102721 A 19920129, Language: English, Database: CAPLUS

Title compds., e.g., I and II [B = H, alkanoyl, alkylsulfonyl, etc.; Q = H, halo, alkoxy, alkyl, etc.; R^1 = H, (cyclo)alkyl, dialkylamino, cation, etc.; R^2 = alkyl; R^3 = (cyclo)alkyl; R^2R^3 = atoms to complete a carbocyclic ring; X, Y, Z = N, O, S, CR^6, CR^7R^8; R^6-R^8 = H, halo, alkoxy, alkyl, etc.; dashed lines = optional bonds] were prepd. Thus, piperonylic acid was converted in 4 steps to 3,4-methylenedioxyphthalic anhydride which was condensed with $Me_2CHCMe(NH_2)CONH_2$ to give, after esterification, I (B = Q = H, R^2 = Me, R^3 = CHM_2, X = Y = O, Z = CH_2, dashed lines = null) (III; R^1 = Me). III (R^1 = furfuryl) (prepn. given) gave 45-100% control of 7 weeds, e.g., 80-90% control of Cyperus rontundus, with moderate damage to corn at 0.125 kg/ha preemergent.



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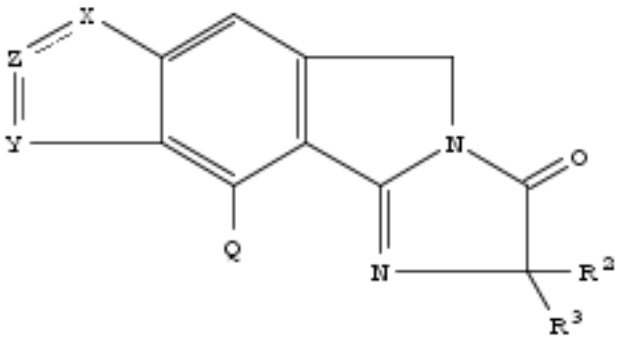


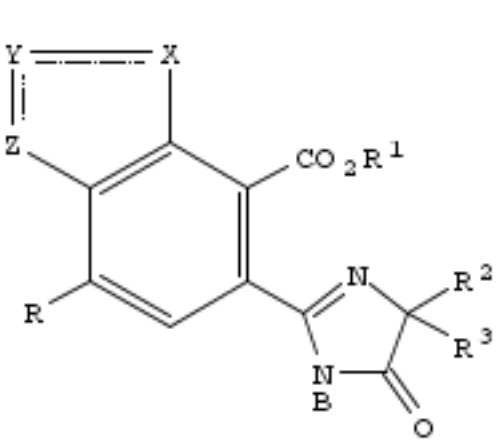
II

~1 Citing

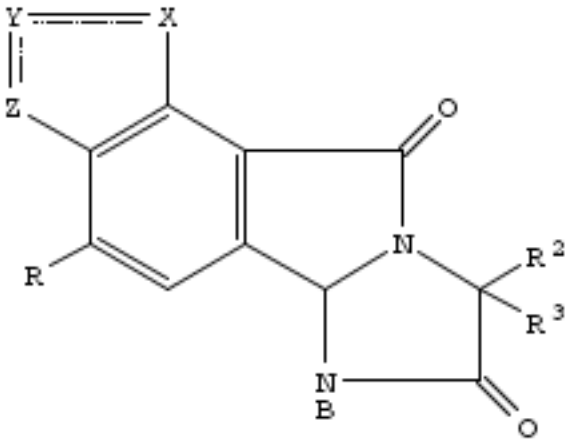
62. Preparation of (2-imidazolin-2-yl)benzazoles and analogs as herbicides

By Doehner, Robert Francis, Jr.
From Eur. Pat. Appl. (1992), EP 474991 A1 19920318, Language: English, Database: CAPLUS

Title compds., e.g., I and II [B = H, alkanoyl, alkylsulfonyl; R = H, halo, alkoxy, alkyl, etc.; R^1 = H, (cyclo)alkyl, alkenyl, alkynyl, cation, etc.; R^2 = alkyl; R^3 = (cyclo)alkyl; R^2R^3 = atoms to complete a (Me-substituted) carbocyclic ring; 1 of X, Y, Z = N or NR^9 and the others = CR^6, CR^7R^8, N, NR^9; R^6-R^8 = H, halo, alkoxy, alkyl, etc.; R^9 = H, alkyl, etc.] were prepd. Thus, 1-ethyl-2-methyl-4,5-benzimidazole dicarboxylic anhydride (prepn. in 5 steps from di-Me 4-acetamidophthalate given) was condensed with α-methylvalinamide and 1 of 2 products cyclized to give title compd. III which at 0.500 kg/ha postemergent gave 80-100% control of 9 weeds with slight effect on soybeans.



I



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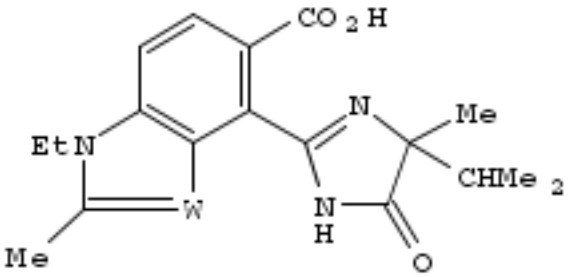


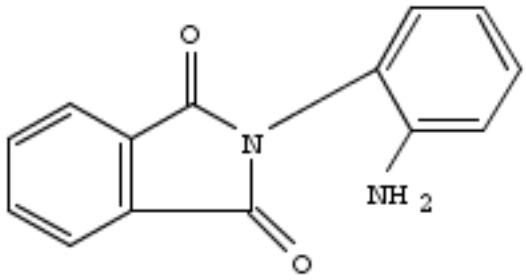
III

~2 Citings

63. Cyclodehydration and hydrolysis of o-amino anilides. V. Kinetics of imidation and hydrolysis of 2'-amino-2-carboxybenzanilide in sulfuric acid solutions at 25°

By Vinnik, M. I.; Shchel'tsyn, V. K.; Sycheva, E. A.; Krasil'nikova, G. S.
From Kinetika i Kataliz (1990), 31(5), 1106-15. Language: Russian, Database: CAPLUS

In 45-94% H_2SO_4, 2-$HO_2CC_6H_4CONHC_6H_4NH_2$-2 (I) is cyclized to imide II and hydrolyzed to o-phenylenediamine and phthalic acid or anhydride. The rate consts. for both reactions increase with increasing H_2SO_4 concn. and reach a max. in the 70-76% range. The rate const. for cyclization is proportional to the relative concn. of the neutral form of I and the function h_0a_{H2O}; the hydrolysis of I proceeds by 2 paths from the neutral form. The complexes of I existing at different H_2SO_4 concns. are identified.



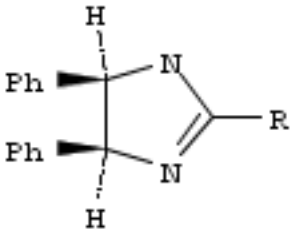
II

~0 Citings

64. Reactions with meso-1,2-diphenylethylenediamine

By Hammouda, Hamdy A.; Abd-Allah, Sanaa O.; Sharaf, Mohie A. F.
From Egyptian Journal of Chemistry (1989), 30(3), 239-47. Language: English, Database: CAPLUS

The reaction of the title compd. (I) with acids RCH_2CO_2H (R = H, Me) gave 2-imidazolines II. The reaction of I with thiosemicarbazide gave as-triazine III. Heating I in the presence of $SOCl_2$ gave thiadiazole IV. 5H-Pyrrolo[1,2-a]imidazol-5-one V was prepd. by heating I and succinic anhydride in the presence of polyphosphoric acid. Heating I with benzil monohydrazone gave triazepine VI. Other reactions of I were studied.



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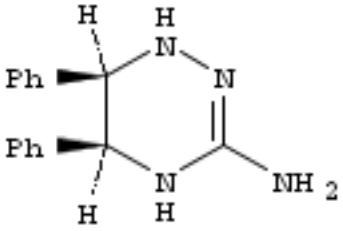
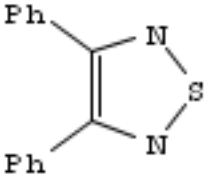


III



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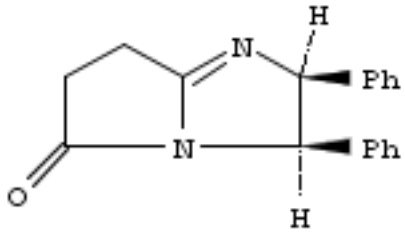
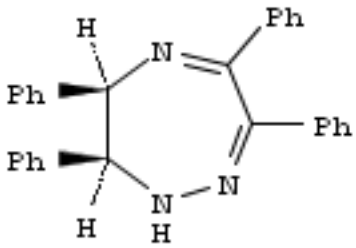


V



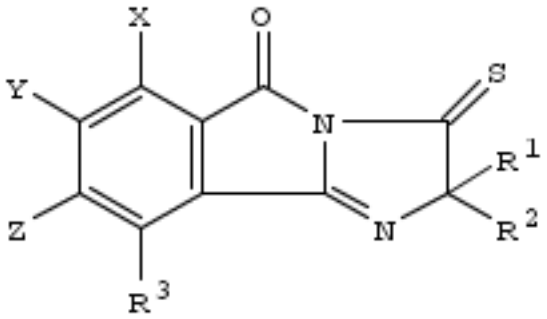
V I

~0 Citings

65. Preparation of herbicidal 3-thio-5H-imidazo(2,1-a)isoindole-3(2H), 5-diones

By Los, Marinus
From U.S. (1990), US 4911747 A 19900327, Language: English, Database: CAPLUS

The title compds. I (R^1, R^2 = C_{1-3} alkyl, cyclopropyl; CR^1R^2 = C_{3-6} cycloalkyl; R^3 = H, halo, C_{1-4} alkyl, CF_3, etc.; X = H, halo, Me; Y, Z = H, halo, C_{1-6} alkyl, CN, NO_2, etc.) and related compds. are prepd. as herbicides. A soln. of o-(4-isopropyl-4-methyl-5-thioxo-2-imidazolin-2-yl)benzoic acid (prepn. given) in THF was treated with dicyclohexylcarbodiimide, to give I (R^1 = Me, R^2 = iso-Pr, R^3 = X = Y = Z = H) (II). Postemergence application of 1 kg II/ha totally controlled barnyard grass (Echinochloa crus-galli), green foxtail (Setaria viridis), wild mustard (Brassica kaber), and other weeds with no damage to rice.



I

~0 Citings

66. Synthesis and spectral studies of platinum complexes with amide-group containing ligands

By Gade, Dayakar; Puri, Lingaiah
From Transition Metal Chemistry (Dordrecht, Netherlands) (1989), 14(3), 203-5. Language: English, Database: CAPLUS, DOI:10.1007/BF01043795

[PtL_2] [HL = 2-(R-substituted-amino)benzoic acid (R = Ac, Bz, Ph), maleanilic acid, malea-1-naphthanilic acid, 2-[1-naphthalenylamino)carbonyl]benzoic acid, 2-(2-aminobenzoylamino)benzoic acid] and PtL^1Cl (HL^1 = 2-[2-aminophenylaamino)carbonyl]benzoic acid, 2-(aminobenzoyl)benzoic acid] were prepd. and characterized by elemental anal., molar cond. measurements, thermal data and IR electronic, and NMR spectra.

~0 Citings

67. Moessbauer and other spectral studies of iron(II) complexes with amide-containing ligands

By Gade, Dayakar; Gubba, Balaswamy; Vadde, Ravindar; Puri, Lingaiah
From Transition Metal Chemistry (Dordrecht, Netherlands) (1987), 12(6), 539-43. Language: English, Database: CAPLUS, DOI:10.1007/BF01023844

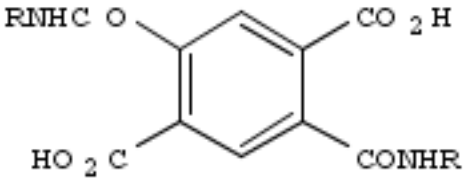
Fe(II)-ligand (1:2) complexes (polydentate ligand = 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, 2-[2-aminobenzoylamino]benzoic acid, 2-[aminocarbonyl]benzoic acid, 2-[(phenylamino)carbonyl]benzoic acid, 2-[aminobenzoyl]benzoic acid and 2-aminobenzanilide) were prepd. and characterized by elemental analyses, cond., magnetic susceptibility and IR, electronic, NMR, and Moessbauer spectral studies. The different modes of ligand chelation and the stereochem. around the metal ion are discussed. The small range of isomer shift values for Fe(II) complexes confirms the similar geometry for all the complexes.

~0 Citings

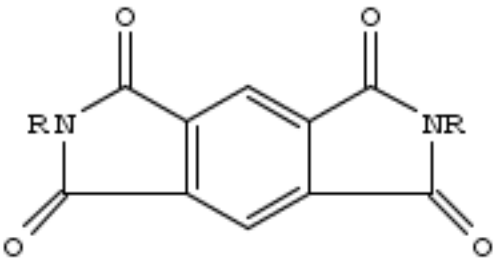
68. The synthesis of pyromellitamic diacids and pyromellitdiimides and their effect on the human serum cholinesterase activity in vitro

By Al-Azzawi, Mohammad J.; Atto, Amir T.; Al-Ahdami, Balqiz W.; Ali, Imad T.
From Journal of Biological Sciences Research (1988), 19(1), 85-93. Language: English, Database: CAPLUS

Eight pyromellitamic diacids I (R = substituted Ph or pyridyl or tetrazyl) were prepd. by reaction of amines with pyromellitic dianhydride and then 4 of them were cyclized by dehydration with the acetic anhydride-sodium acetate system to form the corresponding diimides II (R = substituted phenyl). I and II dose-dependently inhibited cholinesterase of human blood serum.



I



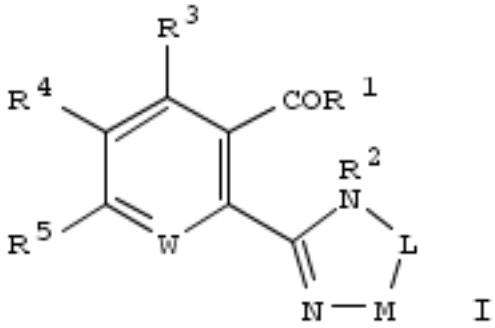
II

~0 Citings

69. Preparation and testing of arylimidazoles as herbicides

By Astles, David Phillip; Flood, Andrew
From Brit. UK Pat. Appl. (1988), GB 2192877 A 19880127, Language: English, Database: CAPLUS

The title compds. (I; R^1 = OR^8, alkyl, alkenyl, alkynyl, cycloalkyl, Ph, furyl, $PhCH_2$; R^2 = H, acyl; R^1R^2 = bond; R^3, R^5 = H, halo, NO_2, cyano, Q; R^4 = H, halo, OH, NO_2, Q; R^6 = alkyl, cycloalkyl; R^7 = alkyl, cycloalkyl, alkenyl, Ph, $PhCH_2$; R^8 = H, salt-forming cation; W = N, CH; one of L, M = CO, the other = CR^6R^7; Q= XYZC; X = cyano, thiol, amino, oximino, etc.; Y = H, alkyl, X; Z = H, alkyl) were prepd. as herbicides. Di-Me 5-ethylpyridine-2,3-dicarboxylate was successively photobrominated with NBS, condensed with NaSMe, sapond. with aq. NaOH, refluxed with Ac_2O to yield an anhydride, and condensed with 2-amino-2,3-dimethylbutyramide to give 2-[(1-carbonyl-1,2-dimethylpropyl)carbonyl]-[5-[1-methylthio)ethyl]nicotinic acid, which was cyclized in 3 M NaOH to give 2-(5-isopropyl-5-methyl-4-oxo-2-imidazolin-2-yl)-5-[1-(methylthio)-ethyl]nicotinic acid (II). At 1 kg/ha preemergent, II gave complete control of Echinochloa crusgalli.



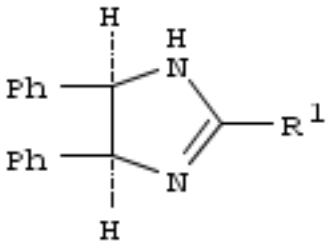
I

~3 Citings

70. Reactions with meso-1,2-diphenylethylenediamine

By Hammouda, Hamdy A.; Abd-Allah, Sanaa O.; Sharaf, Mohie A. F.
From Proceedings of the Pakistan Academy of Sciences (1986), 23(2), 155-66. Language: English, Database: CAPLUS

Dihydroimidazoles I (R^1 = H, Me, SH, NH_2) and II were prepd. A mixt. of meso-$H_2NCHPhCHPhNH_2$ and HOAc was heated to give I (R^1 = Me).



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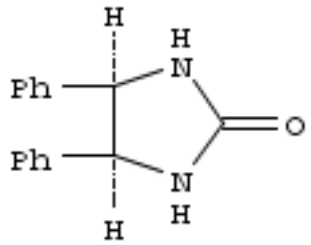


II

~0 Citings

71. Determination of thalidomide and its major metabolites by high-performance liquid chromatography

By Czejka, Martin J.; Koch, Heinrich P.
From Journal of Chromatography, Biomedical Applications (1987), 413, 181-7. Language: English, Database: CAPLUS, DOI:10.1016/0378-4347(87)80225-6

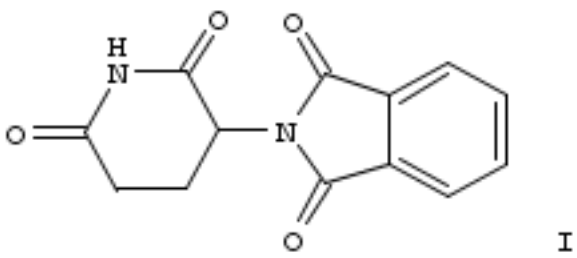
A rapid and sensitive isocratic HPLC assay for the simultaneous and quant. detn. of thalidomide (α-phthalimidoglutarimide)(I) [50-35-1] and its major metabolites from human serum is described. The parent compd. and the metabolites can be efficiently sepd. by reversed-phase chromatog. using tetramethylammonium bromide as an ion-pair-forming reagent. Chromatog. is carried out on either of 2 systems. System 1 consists of LiChrosorb RP-8 pre- and anal. columns, H_2O-MeOH-n-propanol (180:30:20) eluent, and UV detection at 254 nm. System 2 consists of a Spherisorb S5 C_{18} anal. column, H_2O-MeCN (80:20) eluent, and UV detection at 290 nm. The concn.-detection response was linear between 1 and 100 μg/mL for thalidomide, and the coeff. of variation from 10-20 detns. carried out on the same day was 2.3-7.4% for the concn. range 1-20 μg/mL. The detection limit for thalidomide was 0.5-1 μg/mL when a 20-μL loop was used.

~18 Citings

72. Immunological effects of thalidomide. Inactivity of the drug and several of its hydrolysis products in mononucleocyte proliferation tests

By Guenzler, V.; Hanauske-Abel, H. M.; Tschank, G.; Schulte-Wissermann, H.
From Arzneimittel-Forschung (1986), 36(7), 1138-41. Language: English, Database: CAPLUS

The effect of thalidomide (I) [50-35-1] and its 7 hydrolysis products (e.g. II [3343-29-1] and III [30076-84-7]) on the proliferation ([^{3}H]thymidine incorporation) of lectin- and allogeneically stimulated human peripheral blood mononucleocytes was studied. In contrast to expts. reported previously, no inhibition of ^{3}H incorporation was found, regardless of the duration, mode, or timing of stimulation. An HPLC method for the anal. of I hydrolysis products is described.



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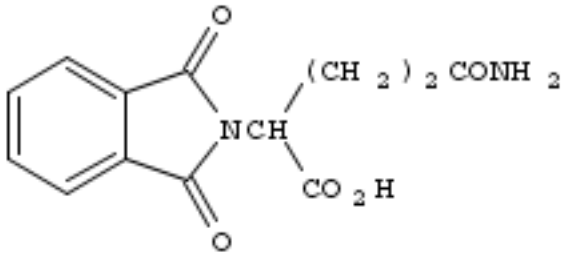

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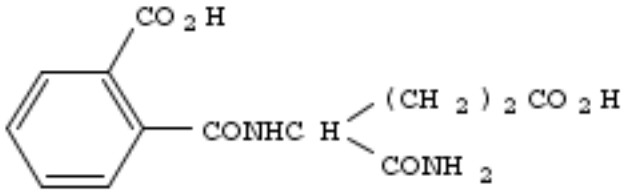


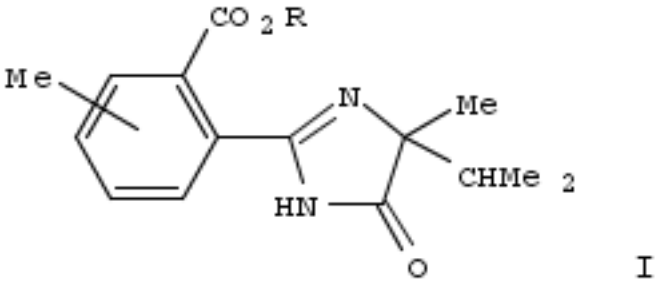
III

~0 Citings

73. Regio and optical isomers of imidazolinyltoluic acids, esters and salts, and their use as herbicidal agents

By Los, Marinus
From Eur. Pat. Appl. (1985), EP 158000 A1 19851016, Language: English, Database: CAPLUS

The imidazolinyltoluic acids I (R = H, Me, alkali metal, NH_4, etc.), as (+)-I and (±)-I mixts. of each the meta and para isomers, and the (+)-I mixt. of both meta and para isomers, are herbicides. Thus, in greenhouse expts., preemergent application of (±)-I (50:50 para/meta mixt.) at 4 kg/ha totally controlled barnyard grass (Echinochloa crus-galli), canary grass (Phalaris), field bindweed (Convalvulus arvensis), and other weeds. I are prepd. by reacting p- or m-toluyl chloride with (±)- or (+)-2-amino-2,3-dimethylbutyramide to form N-(1-carbamoyl-1,2-dimethylpropyl)toluamide, which upon cyclization with a strong base yields 4-isopropyl-4-methyl-2-tolyl-2-imidazolin-5-one. This intermediate is metalated with sec-BuLi and treated with CO_2 to give I.



I

~0 Citings

74. Synthesis and spectral studies of copper(II) complexes with amide group ligands

By Ravindar, V.; Swamy, S. J.; Srihari, S.; Lingaiah, P.
From Polyhedron (1985), 4(8), 1511-18. Language: English, Database: CAPLUS, DOI:10.1016/S0277-5387(00)86991-0

Complexes of Cu(II) with 2-R-substituted benzoic acids (R = 2-aminobenzoyl, acetylamino, benzoylamino, aminocarbonyl, (2-aminophenylamino)carbonyl), 2-aminobenzanilide, maleanilic acid (I), and malea-1-naphthalanilic acid (II) were prepd. and characterized by chem. analyses, molar cond., magnetic susceptibility measurements, thermal data, IR, electronic and ESR spectra. The visible and ESR spectral studies of these complexes (except those of I and II which are tentatively assigned dimeric structures) indicate that they are monomeric having either square planar or distorted octahedral geometry around Cu(II). From the ESR spectra of Cu(II) complexes various parameters were calcd.

~24 Citings

75. Moessbauer and other spectral studies on iron(III) complexes with ligands containing amide group

By Ravindar, V.; Lingaiah, P.; Vithal, M.; Jagannathan, R.
From Indian Journal of Chemistry, Section A: Inorganic, Physical, Theoretical & Analytical (1985), 24A(6), 485-8. Language: English, Database: CAPLUS

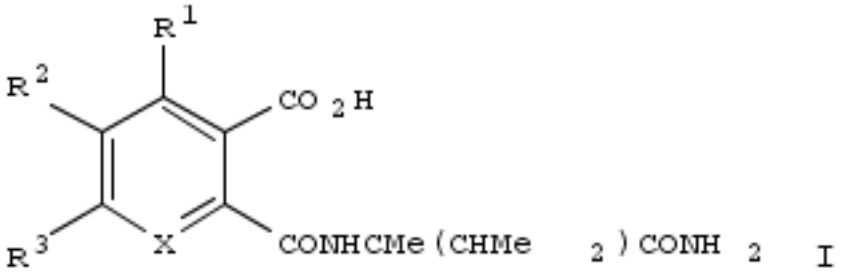
FeL_3 (HL = 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, maleanilic acid, malea-1-naphthanilic acid, 2-(aminocarbonyl)benzoic acid, 2-[(phenylamino)carbonyl]benzoic acid, 2-[(1-naphthalenylamino)carbonyl]benzoic acid), $[FeL^1{}_3](NO_3)_3$ (L^1 = 2-aminobenzanilide) and $[FeL^2{}_2]NO_3$ (HL^2 = 2-[(2-aminophenylamino)carbonyl]benzoic acid, 2-(aminobenzoyl)benzoic acid) were prepd. and characterized from chem. analyses, magnetic susceptibility and IR, NMR, electronic and Moessbauer spectral data.

~0 Citings

76. Substituted and unsubstituted 2-[(1-carbamoyl-1,2-dimethylpropyl)-carbamoyl]-3-quinolinecarboxylic acids, -nicotinic acids, and -benzoic acids

By Gastrock, William Henry; Mason, Timothy Frank; Withers, Gregory Portee
From Ger. Offen. (1985), DE 3441637 A1 19850530, Language: German, Database: CAPLUS

The title compds. [I; X = N, RC; R, R^1 = H, alkyl, halo; R^2 = H, alkyl, alkoxy, F_3C, Cl_3C, F_2CHO, alkylthio, halo, dialkylamino, (un)substituted Ph, PhO; R^3 = H, alkyl, F_3C, Cl_3C, (un)substituted Ph, PhO; R^2R^3 = $(CH_2)_n$, (un)substituted CH:CHCH:CH; n = 3-5] were prepd. Thus, 4-methylphthalic anhydride and $Me_2CHCMe(NH_2)CN$ were stirred at 35-40° in CH_2Cl_2-Me_2SO to give a mixt. of intermediate carbamoylbenzoates which was extd. into aq. NaOH and treated with H_2O_2 at 30° to give I (X = CH, R^1 = R^3 = H, R^2 = Me, R^1 = R^2 = H, R^3 = Me). The reaction mixt. was heated 2-3 h at 80-90° to give 93-94% herbicidal (no data) imidazolylbenzoates II (R^4 = 4-Me, 5-Me).



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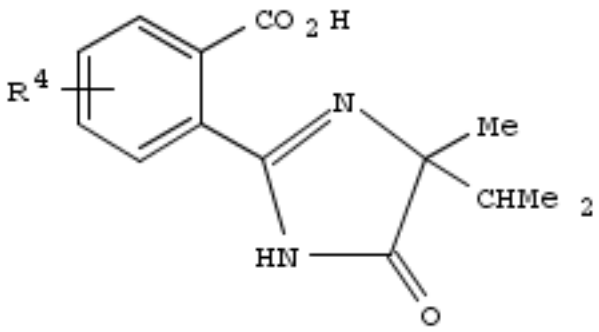


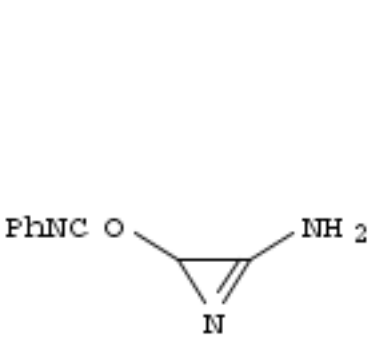
II

~2 Citings

77. Novel method for synthesis of diazocines

By Eremeev, A. V.; Piskunova, I. P.; El'kinson, R. S.
From Khimiya Geterotsiklicheskikh Soedinenii (1985), (6), 848-9. Language: Russian, Database: CAPLUS

Treating azirine I with phthalic anhydride gave 70% diazocine II and 30% PhNHCOCH($CONH_2$)NHC$OC_6H_4CO_2$H-o. Analogously obtained was 79% diazocine III from naphthalic anhydride.



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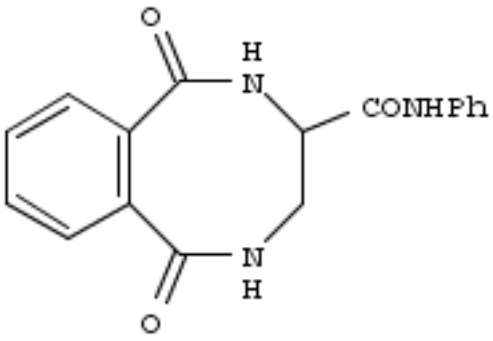

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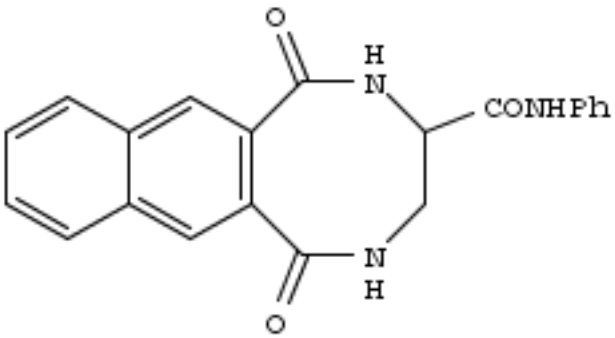


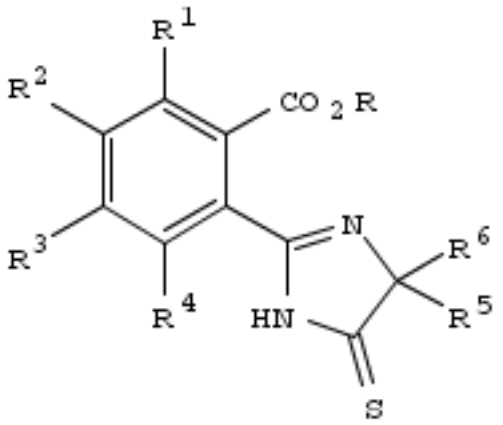
III

~0 Citings

78. 5-Thioxo-2-imidazolinyl benzoic acids, esters, salts and related compounds, and their use as herbicidal agents

By Los, Marinus
From Eur. Pat. Appl. (1985), EP 135711 A1 19850403, Language: English, Database: CAPLUS

(Thioxoimidazolinyl)benzoates I and their cyclized derivs. II [R = (un)substituted alkyl, alkenyl, alkynyl, cycloalkyl, cation; R^1 = H, halogen, Me; R^2, R^3 = H, halo, (un)substituted alkyl, alkoxy, alkylthio, phenoxy, etc.; R^2R^3 = $(CH_2)_n$, (un)substituted CH:CHCH:CH; R^4 = H, halo, alkyl, alkylthio, alkoxy, CF_3, NO_2, OCF_3, $OCHF_2$, OCF_2CHF_2; R^5, R^6 = alkyl, cyclopropyl; R^7 = H, OH, alkoxy, amino, R^8 = H; R^7R^8 = bond; X = C:S, X^1 = CR^5R^6; X = CR^5R^6, X^1 = C:S; n = 2-4] were prepd. Thus, phthalic anhydride was aminated with $H_2NCMe(CHMe_2)CSNH_2$ to give 2-$HO_2CC_6H_4CONHCMe(CHMe_2)CSNH_2$, which cyclized to give (thioxoimidazolinyl)benzoate III (R = H). At 0.125 kg/ha pre-emergence, III (R^2 = $PhCH_2$) gave 100% kill of quackgrass (Agropyron repens), whereas barley was undamaged.



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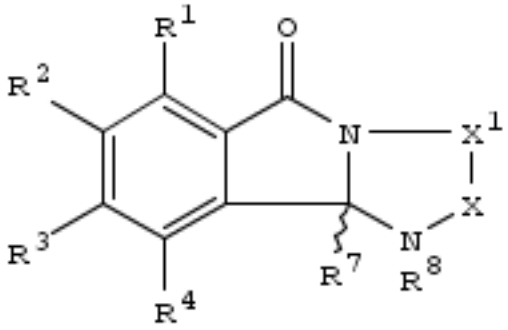

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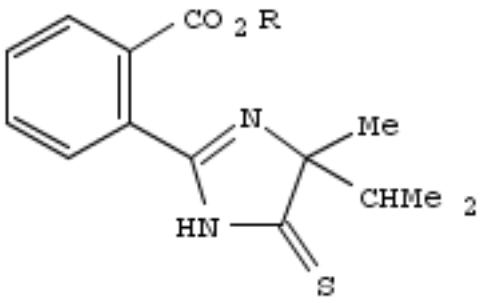


III

~1 Citing

79. Effect of amide group ligands and their metal complexes on pathogenic fungi

By Ravindar, V.; Lingaiah, P.
From Current Science (1984), 53(19), 1032-4. Language: English, Database: CAPLUS

The fungicidal activity of the ligands maleanilic acid (MA) [555-59-9], malea-1-naphthalanilic acid (MNA) [6973-77-9], 2-(aminocarbonyl)benzoic acid (ACBA) [88-97-1], 2-[(phenylamino)carbonyl]benzoic acid (PACBA) [4727-29-1], 2-[(1-naphthalenylamino)carbonyl]benzoic acid (NACBA) [132-66-1], 2-[(2-aminophenylamino)carbonyl]benzoic acid (APACBA) [7297-65-6], 2-aminobenzanilide (ABn) [4424-17-3], and 2-(aminobenzoyl)benzoic acid (ABBA) [1147-43-9] and their Pd(II) complexes, as well as APACBA complexes of Co(II), Ni(II), and Cu(II) against Drechslera rostrata, Curvularia lunata, and Aspergillus niger depended on the type of metal ion and on the type of ligand. Pd(II) complexes exhibited higher fungistatic activity than did the free ligands and Dithane M-45. The fungistatic activity of Pd(II) chelates followed this order: APACBA > ABBA > ABn > ACBA > PACBA > NACBA > MA > MNA. APACBA complexes showed higher fungistatic activity than did the free ligand, with Pd(II) complex exhibiting max. activity and Co(II) complex min. activity. The toxicity of metal complexes followed the order Pd > Cu > Ni > Fe > Co.

~1 Citing

80. Studies on ruthenium(II) complexes with ligands containing the amide group

By Ravindar, V.; Lingaiah, P.; Reddy, K. Veera
From Inorganica Chimica Acta (1984), 87(1), 35-40. Language: English, Database: CAPLUS, DOI:10.1016/S0020-1693(00)83617-8

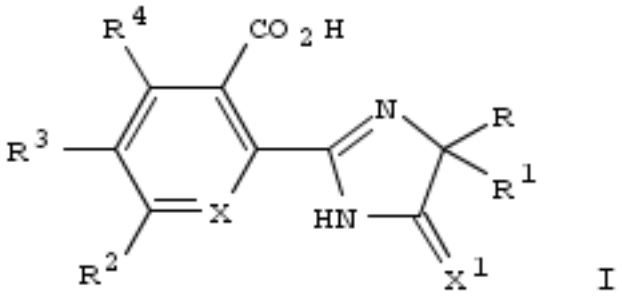
$RuCl_2(DMSO)_4$ reacted with various amide-group-contg. ligands to give $Ru(DMSO)_2L_2$ (HL = $RNHC(O)R^1$, R = o-$(HO_2C)C_6H_4$, R^1 = CH_3, C_6H_5; R = C_6H_5, R^1 = CH:$CH(CO_2H)$; R = H, C_6H_5, naphthyl, R^1 = o-$(HO_2C)C_6H_4$), RuL_2 (R = o-$(HO_2C)C_6H_4$, R^1 = o-$H_2NC_6H_4$; R = o-$H_2NC_6H_4$, R^1 = o-$(HO_2C)C_6H_4$), $RuL^1{}_2$ (HL^1 = 2-(2-aminobenzoyl)benzoic acid), and $RuCl_2L^2{}_2(DMSO)_2$ (HL^2 = $H_2NC(O)C_6H_4NH_2$-o) which were characterized by elemental anal., elec. cond., and electronic, IR, and ^1H NMR spectral methods.

~4 Citings

81. 2-(5,5-Disubstituted-4-oxo-2-imidazolin-2-yl)nicotinic acids and quinoline-3-carboxylic acids

By Barton, Jerry Michael; Long, Don Wesley; Lotts, Kenneth Dale
From Eur. Pat. Appl. (1983), EP 95105 A2 19831130, Language: English, Database: CAPLUS

Herbicidal (no data) imidazoles I [X = N, CH; X^1 = O, S; R = alkyl; R^1 = alkyl, cycloalkyl; RR^1 = alkylene; R^2 = H, alkyl, CF_3, CCl_3, Ph, substituted Ph; R^3 = H, halogen, alkyl, alkoxy, CF_3, CCl_3, CHF_2O, amino, alkylthio, NO_2, Ph, OPh, substituted Ph, OPh; R^4 = H, alkyl; R^2R^3 = alkylene, (un)substituted CH:CHCH:CH] were prepd. Thus, 2,3-pyridinedicarboxylic anhydride was aminolyzed with $H_2NCMe(CHMe_2)CONH_2$ to give a mixt. of pyridinemonocarboxamides which were treated with NaOH to give II and its regioisomer.



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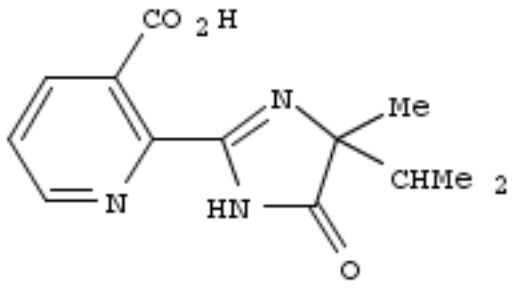


II

~8 Citings

82. Structural studies of palladium complexes with ligands containing the amide group

By Vadde, Ravindar; Jagannatha, Swamy S.; Somu, Srihari; Puri, Lingaiah
From Transition Metal Chemistry (Dordrecht, Netherlands) (1984), 9(3), 103-6. Language: English, Database: CAPLUS, DOI:10.1007/BF00618585

PdL_2 (HL = 2-(acetylamino)benzoic acid, 2-(benzoylamino)benzoic acid, 2-hydroxybenzanilide, 2-mercaptobenzanilide, maleanilic acid, 2-(aminocarbonyl)benzoic acid, 2-[(phenylamino)carbonyl]benzoic acid, 2-[(1-naphthalenylamino)carbonyl]benzoic acid, salicylanilide, 2-(aminobenzoyl)benzoic acid), PdL^1Cl (HL^1 = 2-[2-aminobenzoylamino]benzoic acid, 2-[(2-aminophenylamino)carbonyl]benzoic acid), and $PdL^2{}_2Cl_2$ (L^2 = 2-aminobenzamide) were prepd. The complexes were characterized by chem. analyses, molar cond. measurements, thermal data, and IR, electronic and NMR spectra.

~3 Citings

83. Transformations of phthalic acid N-o-aminoanilide in acid media

By Mukhina, O. A.; Nechaev, P. P.; Zaikov, G. E.
From Vysokomolekulyarnye Soedineniya, Seriya B: Kratkie Soobshcheniya (1982), 24(10), 778-80. Language: Russian, Database: CAPLUS

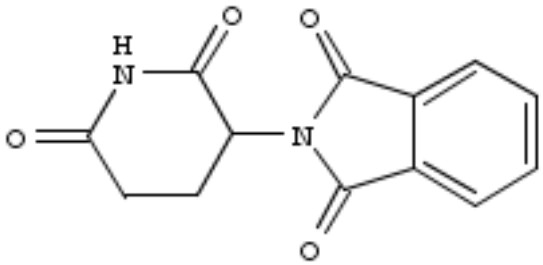
phthalic acid N-o-aminoanilide (I) [7297-65-6], used as a model compd. for polyamic acid, underwent hydrolysis in acidic H_2O-MeOH solns. to form o-phenylenediamine [95-54-5] and phthalic anhydride [85-44-9], accompanied by the intramol. cyclization to form 1,2-benzoylbenzimidazole [2717-05-7]. The activation energy of hydrolysis and cyclization of I was 122 kJ/mol. The pH-dependencies of the rate consts. of hydrolysis and cyclization of I were given.

~0 Citings

84. Studies on the hypothetical relationship of thalidomide-induced embryopathy and collagen biosynthesis

By Flohe, L.; Draeger, E.; Frankus, E.; Graudums, I.; Guenzler, W. A.; Helm, F. C.; Kuutti-Savolainen, E. R.
From Arzneimittel-Forschung (1981), 31(2), 315-20. Language: English, Database: CAPLUS

The hypothesis that thalidomide (I) [50-35-1] or a metabolite thereof exerts its teratogenic effects by interfering with embryonal protocollagen hydroxylation was checked by in vitro and in vivo studies. In vitro activity of prolyl hydroxylase [9028-06-2] of 3 different sources (mouse, chicken embryo, and man) and lysyl hydroxylase [9059-25-0] (mouse) were only inhibited by high concns. of thalidomide, 6 putative metabolites of thalidomide and 2 structurally related compds., EM 8 (3-(2,3-dihydro-1,1-dioxido-3-oxo-1,2-benzisothiazol-2-yl)-2,6-dioxopiperidine) [16477-31-9] and EM 12 (3-(1,3)-dihydro-1-oxo-2H-isoindol-2-yl)-2,6-dioxopiperidine) [26581-81-7]. No correlation was found between the slight in vitro inhibition of protocollagen hydroxylation and the teratogenic potential of these compds., as derived from studies in whole animals or organ culture systems, resp. In vivo total collagen synthesized during the organo-genetic period of rabbit fetuses appeared to be slightly depressed by thalidomide treatment (20, 40, and 80 mg/kg, twice a day) between day 7-16 of pregnancy. Concomitantly a dose-dependent incidence of malformations was obtained. However, the presence of malformations was not assocd. with a particularly low collagen content of malformed fetuses. Treatment with high dosages of supidimide (80 mg/kg, twice a day) similarly affected total fetal collagen formation, but did not induce any malformation. The hydroxyproline content of fetal collagen did not differ between thalidomide treated and control groups. Thus, the minor effects of thalidomide or its metabolites on collagen biosynthesis cannot explain the induction of fetal malformations.



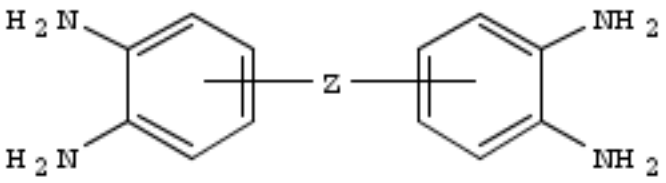
I

~9 Citings

85. Soluble polyimides based on poly(o-amino-o-carboxy)amides

By Korshak, V. V.; Rusanov, A. L.; Batirov, I.; Katsarava, R. D.; Niyazi, F. F.
From Faserforschung und Textiltechnik (1978), 29(11-12), 649-59. Language: German, Database: CAPLUS

High-mol.-wt. poly(o-amino-o-carboxy)amides are prepd. by the reaction of compd. I (Z = O, CH_2, a bond, CO, SO_2, or p-OC_6H_4O) with pyromellitic dianhydride [89-32-7] or compd. II (Z = CO, O, or SO_2) in DMF. Treatment of the reaction solns. contg. the poly(o-amino-o-carboxy)amides with pyridine-Ac_2O [108-24-7] gives poly(o-acetamido)imides, e.g., with repeating units III, which are sol. in org. solvents. Treatment of the poly(o-amino-o-carboxy)amides with phthalic anhydride [85-44-9] or another arom. anhydride gives poly(o-carboxy)amides with carboxamide side groups, and these polymers are cyclized with pyridine-Ac_2O to prep. polyimides which have pendant arylimido groups, have good thermal stability (10% wt. loss at 430-80° in air), and are sol. in org. solvents. Model compds. (15) are also prepd. by the reactions of o-phenylenediamine [95-54-5] and the tetraamines I with arom. anhydrides.



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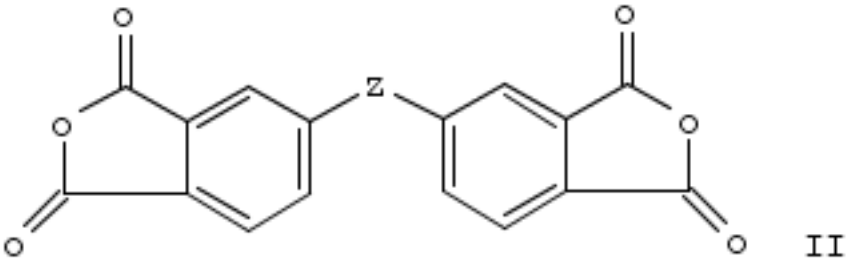

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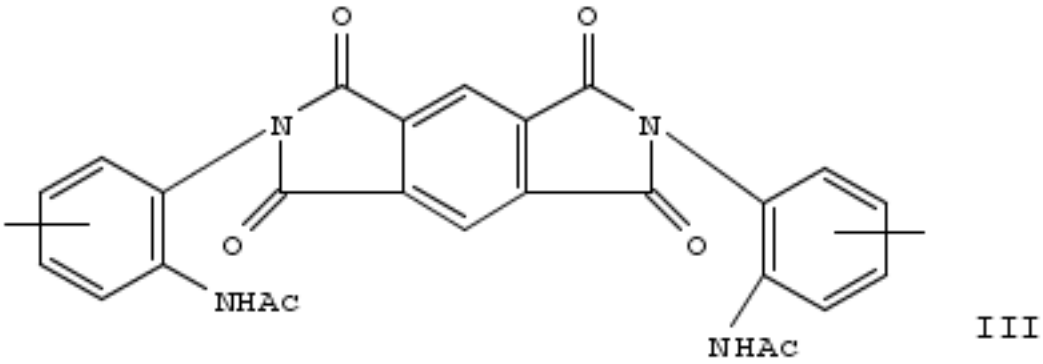


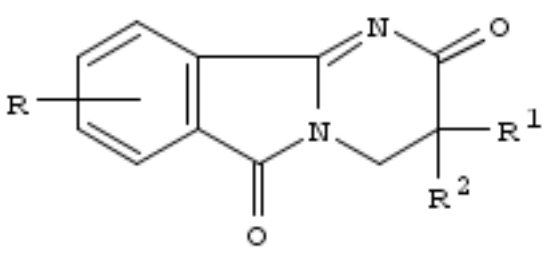
III

~3 Citings

86. Imidazoisoindolediones

By Los, Marinus
From Ger. Offen. (1978), DE 2700270 A1 19780713, Language: German, Database: CAPLUS

The herbicidal compds. I (R = H, Me, NO_2, Cl, OMe, SMe; R^1 = C_{1-4} alkyl; R^2 = C_{1-6} aliph. group, C_{3-6} cycloalkyl, Ph, ClC_6H_4, $PhCH_2$) were prepd. Thus, $Me_2CHCMe(CN)NH_2$ reacted with phthalic anhydride to give 2-$HO_2CC_6H_4CONHCMe(CN)CHMe_2$, which was heated with $SOCl_2$ in CH_2Cl_2 to give II (R^2 = CN). The nitrile was stirred with H_2SO_4 in CH_2Cl_2 to give II (R^2 = $CONH_2$), which was heated with PhMe with removal of H_2O to give I (R = H, R^1 = Me, R^2 = $CHMe_2$), which at 0.14 kg/ha gave complete destruction of Brassica kaber.



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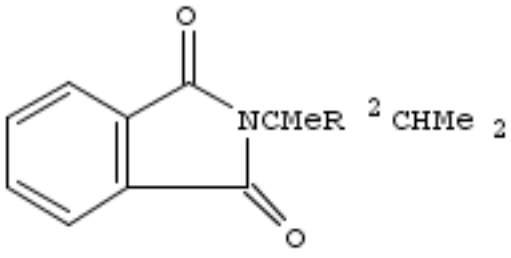


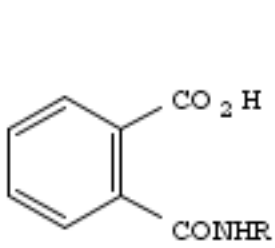
II

~0 Citings

87. Some effective factors in the formation of normal and isoimides

By Awad, William I.; Wasfi, Adel S.; Ewad, Mohammed J. S.
From Journal of the Iraqi Chemical Society (1977), 2(1), 5-16. Language: English, Database: CAPLUS

The Ac_2O-NaOAc dehydration of phthalamic acids I (R = Ph, tolyl, $MeOC_6H_4$, $H_2NC_6H_4$, ClC_6H_4, $O_2NC_6H_4$) yielded imides II. Isoimides III were obtained by the treatment of I with N,N'-dicyclohexylcarbodiimide. Similarly prepd. were diphenimides IV (R = Ph, o-tolyl, p-tolyl); the resp. isoimides were not obtained.



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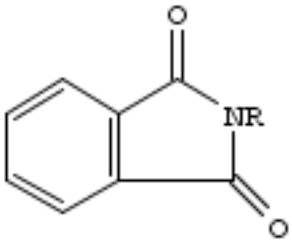

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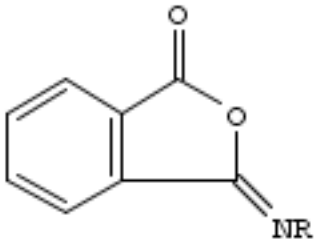

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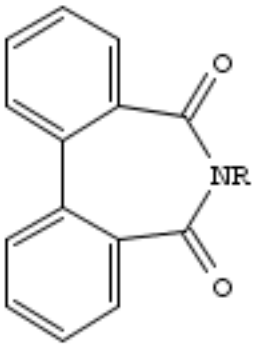

IV

~4 Citings

88. Synthesis and study of soluble poly(o-amido) imides

By Korshak, V. V.; Rusanov, A. L.; Katsarava, R. D.; Niyazi, F. F.
From Vysokomolekulyarnye Soedineniya, Seriya A (1973), 15(12), 2643-9. Language: Russian, Database: CAPLUS

Amino carboxy polyamides with partial structure I were heated with 1:1 complexes of Ac2O or AcCl with a tertiary amine to give acetamido polyimides of partial structure II. The polyamides were prepd. by reaction of 1 of 4 dianhydrides, including 4,4'-oxybis(phthalic anhydride) [1823-59-2], with 1 of 5 tetramines, including bis(3,4-diaminophenyl) ether [2676-59-7]. The presence of the AcNH groups enhanced the soly. of the polyimides in org. solvents. In thermogravimetric studies, typically bis(3,4-diaminophenyl) ether-pyromellitic dianhydride copolymer [25569-64-6] was stable to .sim.400.deg. in an inert atm., at which point further cyclization occurred with elimination of AcOH to give a polyimidazopyrrolone or poly(benzoylenebenzimidazole). The course of the reaction was shown by ir and NMR spectroscopic studies and by the reaction of model compds., such as the cyclization of N-(o-aminophenyl)phthalamic acid [7297-65-6] to N-(o-acetamidophenyl)phthalimide [6543-35-7].

~0 Citings

89. Teratologic determination of hydrolyzed products of thalidomide

By Meise, W.; Ockenfels, H.; Koehler, F.
From Experientia (1973), 29(4), 423-4. Language: German, Database: CAPLUS

Of the 12 hydrolysis products of thalidomide (I) [50-35-1] only the 3, 2-phthalimido-DL-glutaramic acid [7607-72-9], 4-phthalimido-DL-glutaramic acid [4292-56-2], and 2-phthalimido-DL-glutaric acid [6349-98-0], contg. the phthalimide moiety were teratogenic. N-(o-carboxybenzoyl)-DL-glutamic acid imide [40548-80-9] and DL-glutamic acid imide HCl [24666-56-6] did not produce fetal malformations after their i.p. administration to pregnant mice in solns. contg. 25% Tween 20.

~10 Citings

90. Amino acids. 61. Correlation of the configurations of (3S)-amino-2-methylbutanoic acids with that of 2,3-diaminobutane

By Bregant, N.; Zarak, B.
From Bulletin Scientifique, Section A: Sciences Naturelles, Techniques et Medicales (Zagreb) (1972), 17(7-8), 218-19.
Language: English, Database: CAPLUS

(3S)-H2NCHMeCHMeCO2H (I) with sp. rotation of +4° was chem. correlated with meso-H2NCHMeCHMeNH2 (II), showing its threo configuration. Thus, I was converted with phthalic anhydride to the phthalimido deriv. which underwent Schmidt degrdn. to give o-HO2CC6H4CONHCHMeCHMeNH2. The latter was hydrolyzed by HI to yield II.

~0 Citings

91. Cyclization of monomeric and polymeric aminoamido acids

By Korshak, V. V.; Rusanov, A. L.; Katsarava, R. D.
From Vysokomolekulyarnye Soedineniya, Seriya A (1972), 14(9), 1917-23. Language: Russian, Database: CAPLUS

The cyclization of N-(o-aminophenyl)phthalamic acid (I) [7297-65-6] [model of the title polymers (II)] at 180.deg. gives besides N-(o-aminophenyl)phthalimide (III) [4506-62-1] also 30-50% 2-(o-carboxyphenyl)benzimidazole (IV) [16529-06-9] and 7-10% 11-oxo-11H-isoindolo[2,1-a]benzimidazole (V) [2717-05-7]. The presence of o-carboxyphenyl groups in II cyclized in the solid phase is not obsd. This suggests that the cyclization of I proceeds by a different mechanism than the cyclization of II. The cyclization of I in DMF or AcNMe2 gives different amts. of III-V, showing the effects of the reaction kinetics on the distribution of products. The lack of o-carboxyphenyl groups in II is caused by kinetic factors. A complex mechanism is proposed for the formation of IV and V from III.

~2 Citings

92. Synthesis and teratological testing some thalidomide metabolites

By Meise, W.; Koehler, F.
From Pharmazie (1972), 27(6), 418-19. Language: German, Database: CAPLUS

N-[o-Carboxybenzoyl]-DL-glutamine and -isoglutamine were synthesized, and their teratol. activity was compared to that of N-phthaloyl-DL-glutamine and -isoglutamine. Teratol. activity is lost when the phthalimido ring is cleaved.

~0 Citings

93. Poly(imidazopyrrolone) model compounds

By Young, Philip R.
From Journal of Heterocyclic Chemistry (1972), 9(2), 371-8. Language: English, Database: CAPLUS, DOI:10.1002/jhet.5570090232

The model reactions between phthalic anhydride (I) [85-44-9] and o-phenylenediamine (II) [95-54-5] were studied under conditions analogous to the polymn. and postcyclization of dianhydrides with bis(o-diamines) to form poly(imidazopyrrolone). Thus, I was condensed with II in DMF to give 80% N-(o-aminophenyl) phthalamic acid (III) [7297-65-6] which when stored in aq. DMF gave a 2:1 mixt. of N-(o-aminophenyl) phthalimide (IV) [4506-62-1] and 2-(o-carboxyphenyl) benzimidazole (V) [16529-06-9]. Sublimation of IV at 200.deg. gave 11H-isoindolo[2,1α] benzimidazol-11-one (VI). O-phenylenebibenzimidazole and N,N'-diphthaloyl-o-phenylenediamine were obtained as by-products of the melt reaction of I and II. When 10% III in DMF was heated at 152-4.deg. it gave IV and V. When III was melted at 155.deg. it gave 36% V, 19% VI, and a benzimidazole-amide-imide (VII) [35411-16-6]. The IR spectra of the model compds. are given.

~11 Citings

94. Thalidomide. Plant vegetation? Phytopharmacological investigation of thalidomide, its metabolites, and structure related compounds

By Koch, H.
From Scientia Pharmaceutica (1971), 39(4), 209-47. Language: German, Database: CAPLUS

The effect of thalidomide (I) [50-35-1] and its initial hydrolysis product, α-phthalamidoglutarimide (II) [6139-18-0], 11 other metabolites of I, and 19 structurally similar compds. on the growth rate of Lepidium sativum sprouts were studied. I and II had no effect whereas the remaining metabolites and structurally related compds., e.g. N-(1-carboxypropyl)phthalimide (III) [35340-62-6] and N-benzoyl-γ-aminobutyric acid (BzNH[CH2]3CO2H) [35340-63-7] acted as auxins at low concns. (10-4M). The most effective was III which produced a 90% av. increase in plant length. This exptl. report also included a review with 333 refs. on the suitability of plant tests for screening potential teratogenic substances.

~5 Citings

95. Curable compositions of epoxy resins and 4,6-bis(substituted carbamoyl)isophthalic acid

By Moran, Raymond M., Jr.; Kretow, Robert P.
From U.S. (1971), US 3627704 A 19711214, Language: English, Database: CAPLUS

Seven carbamoyl isophthalic acid compds. were prepd. and used for crosslinking epoxy resins and producing high-temp. adhesives, castings, and moldings. Thus, a blend of 100 parts bisphenol A-epichlorohydrin resin [25068-38-6] and 29 parts 4,6-bis[(2-aminophenyl)carbamoyl]isophthalic acid (I) [7297-68-9] was cured at 150.deg. in 269 sec. After 1 year, the blend had a gel-time 226 sec at 150.deg.. 1,2-Diaminobenzene and pyromellitic dianhydride reacted in DMF to give I. A high-temp. adhesive was prepd. from 100 parts methylenedianiline tetraepoxide and 30 parts I.

~0 Citings

96. Curable epoxy resin compositions containing phthalamic acid-type curing agents

By Moran, Raymond M., Jr.; Kretow, Robert P.
From U.S. (1971), US 3627730 A 19711214, Language: English, Database: CAPLUS

Epoxy resins were cured by using phthalamic acid latent hardeners at elevated temps. to form adhesive and coating compns. Thus, 100 parts liq. epoxy from bisphenol A-epichlorohydrin copolymers [25068-38-6] contg. 50 parts phthalamic acid [88-97-1] hardener was cured at 150.deg. and had gel time 19 min. Similar epoxies contg. 44 parts N,N'-bis(2-aminophenyl)-3,3'-(acetoxy-1,3-glycerol)bis(trimellitic acid amide) (I) [34376-38-0] and 63 parts phthalanilic acid [4727-29-1] had gel times 4 and 13 min, resp.

~0 Citings

97. Embryotoxic activity of N-phthaloyl-DL-isoglutamine

By Meise, W.; Koehler, F.
From Zeitschrift fuer Naturforschung, Teil B: Anorganische Chemie, Organische Chemie, Biochemie, Biophysik, Biologie (1971), 26(10), 1081-2. Language: German, Database: CAPLUS

N-phthaloyl-DL-isoglutamine (I) [2820-44-2], the main hydrolysis product of thalidomide [50-35-1], was prepd. and a soln. of I in physiol. saline and Tween 20 was injected i.p. into mice on day 9 after artificial fertilization. Doses of 50 or 200 mg/kg caused 12.8 or 43.3% embryo resorptions and 0 or 10.5%, resp., deformations. DL-isoglutamine [328-48-3] was not embryotoxic even at 800 mg/kg.

~1 Citing

98. Effect of the chemical imperfection of macromolecules on the thermal stability of pyrrones

By Korshak, V. V.; Doroshenko, Yu. E.; Khomutov, V. A.; Fedorova, R. D.
From Doklady Akademii Nauk SSSR (1971), 200(6), 1361-4 [Chem]. Language: Russian, Database: CAPLUS

The thermal decompn. of 3,3'-diaminobenzidine-pyromellitic dianhydride copolymer (I) [25266-51-7] starts in the air or in He at 100.deg., but the poly(benzoylenebenzimidazole) (II) (prepd. by the dehydration-cyclization of I in vacuum at 350.deg.) begins to lose CO2 at about 350.deg.. II contains some uncyclized groups, as in I, which are 1st to decompose. The evolution of CH4 and H begins at 400.deg. for both I and II. Higher hydrocarbons are formed at .sim. 500.deg.. DTA curves of I, II, and model compds. are shown. A decompn. mechanism is proposed.

~0 Citings

99. Thin-layer chromatography of thalidomide and its hydrolysis products

By Pischek, G.; Kaiser, Erich; Koch, Heinrich
From Mikrochimica Acta (1970), (3), 530-5. Language: German, Database: CAPLUS

Thin-layer chromatographic Rf values are given for thalidomide (I) and 12 I hydrolysis products on silica gel G and GF254 plates with 5 mixed developing solvents. The spots were detected fluorometrically or with iodine-CHCl3, bromocresol green, ninhydrin, or Fe-hydroxylamine reagents.

~0 Citings

100. Intermediates for the preparation of thermally stable polymers. IV. Symmetrical quinoxalines and benzoylbenzimidazoles

By Preston, Jack; De Winter, W. F.
From Journal of Heterocyclic Chemistry (1970), 7(2), 433-4. Language: English, Database: CAPLUS, DOI:10.1002/jhet.5570070236

2,2'-Bis(p-nitrophenyl)-7,7'-biquinoxaline was prepd. from p-O2NC6H4COCHO and 3,3'-diaminobenzidine in AcNMe2. 4,4'-Bis(6-nitroquinoxalin - 2-yl)diphenyl ether and 2,2'-p-phenylenebis(6-nitroquinoxaline) were similarly prepd. in dioxane and reduced to the corresponding diamines. 4-Nitro-o-phenylenediamine and 3,3',4,4'-benzophenonetetracarboxylic dianhydride were condensed to give 4,4'-dicarboxy - 3,3'-(2-amino-5-nitrophenylcarbamoyl)benzophenone, which was cyclized in polyphosphoric acid to give (I). Attempts to reduce I failed. (II) was similarly prepd. and was converted to its acid chloride.

~1 Citing

101. Anionic dispersions of polyurethanes

No Inventor data available
From Fr. (1969), FR 1559356 19690307, Language: French, Database: CAPLUS

Reproducible, light-stable, aq. anionic dispersions of polyurethanes contg. 0.05-8% CO2- groups are prepd. without emulsifiers by the polyaddn. of high- and (or) low-mol.-wt. compds. having several reactive H atoms, diisocyanates, and diamino carboxylic acid salts of structure [H2N(CH2)nCHR1CHR] 2NCOR2CO2M, where n = 0 or 1, R and R1 = H or C1-6 alkyl, R2 = alkylene, phenylene, or cyclohexylene, and M = Li, Na, K, Rb, Cs, NH4 (or substituted ammonium), followed by transformation into an aq. dispersion. To vary the water resistance, feel, surface, and brightness of products prepd. by using these dispersions, the reactive-H compds. may be replaced at least partially by carbofunctional polysiloxanes having reactive H atoms and mol. wt. 300-20,000. Thus, 123 g. (NCCH2CH2)2NH was added dropwise at 10-20° to 100 g. succinic anhydride in 200 ml. EtOH, and the mixt. was stirred 1 hr. at 20-40° to give 159 g. (NCCH2CH2)2NCOCH2CH2CO2H (I), m. 112-14°. I (111.5 g.) was dissolved in 600 ml. MeOH, 20 g. NaOH added to neutralize the acid, 150 ml. MeOH added, the Na salt hydrogenated at 80° in the presence of 25 g. Raney Co under 150 atm. H, the pressure removed, the catalyst sepd., and the clear-yellow soln. evapd. to give 134 g. (H2NCH2CH2CH2)2NCOCH2CH2CO2Na (II). 2-NaO2CC6H4CON(CH2CH2CH2NH2)2 (III) was prepd. similarly. Adipic acid-ethylene glycol polyester (IV) (250 g., OH index 56) was dehydrated at 120° during 30 min., the product reacted for 2 hrs. at 120° with 38 g. OCN(CH2)6NCO, the preaddn. product cooled to 55° and dissolved in 700 ml. Me2CO, 15.8 g. II in 80 ml. water added, 450 ml. water added, the mixt. stirred until homogeneous, and Me2CO distd. under vacuum at 55° to give a dispersion contg. 44% solid and 0.5% residual Me2CO. Other polyurethane dispersions were prepd. from the following reactants: adipic acid-1,6-hexanediol-neopentyl glycol polyester, OCN(CH2)6NCO, III; IV, methylcyclohexane diisocyanate (V), II; polypropylene glycol, V, II; HOCH2SiMe2O[SiMe2O]12SiMe2CH2OH, OCN(CH2)6NCO, III.

~0 Citings

102. Polypeptides. XI. Tetrazole analogs of the C-terminal tetrapeptide amide sequence of the gastrins

By Morley, John S.
From Journal of the Chemical Society [Section] C: Organic (1969), (5), 809-13. Language: English, Database: CAPLUS, DOI:10.1039/j39690000809

The synthesis is described of 2 analogs of N-benzyloxycarbonyl-L-tryptophyl-L-methionyl-L-aspartyl-L-phenylalanine amide (a physiol. active deriv. of the C-terminal tetrapeptide amide sequence of the gastrins) wherein the aspartyl β-carboxy group or the terminal carboxamide group of the tetrapeptide amide are replaced by a tetrazol-5-yl residue. The optically active amino acid analogs (carboxy replaced by tetrazol-5-yl) required in these syntheses were prepd. without special difficulties, and could be utilized in peptide synthesis without protection of the tetrazole unit.

~12 Citings

103. Synthesis and investigation of polybenzimidazopyrrolones containing ether linkages in the main chains

By Korshak, V. V.; Rusanov, A. L.; Katsarava, R. D.
From Doklady Akademii Nauk SSSR (1968), 182(6), 1327-30. Language: Russian, Database: CAPLUS

Poly[(p-phenylene oxide)benzimidazo-pyrrolones] are synthesized, the structures are elucidated using ir spectra, and thermal properties of the compds. are studied. 3,3',4,4'-Tetraaminodiphenyl ether or hydroquinone 3,3'-4,4'-tetraaminodiphenyl ether (0.004M) was cooled under argon to -20 to -30°, and 10-12 ml. HCONMe2 was added. To the soln. was added a soln. of 0.825 g. pyromellitic dianhydride in 10 ml. HCONMe2, and the mixt. allowed to react. The ir spectra of the obtained polymers showing absorptions at 1640, 1540, and 1305 cm.-1, were representative of a poly(aminocarboxamide), when compared with the model compd. N-(o-aminophenyl)-phthalamic acid. The polymer was heated at 150-60° for 5 hrs. to give a yellow-orange polymer. The ir spectrum was compared with those of the model compds. N-(o-aminophenyl)phthalimide and 2-(o-carboxyphenyl)benzimidazole to show that the polymer had a poly(aminoamide) structure. Further heating of the polymer to 300-30° for 10 hrs. gave a benzimidazolepyrrolone (I, X = O or p-OC6H4O); the ir spectra was compared with that of 1,2-dibenzoylbenzimidazole. The compds. have tear strength 1000-1100 kg./cm.2 and elongation 3-4%. They are sol. on heating in concd. H2SO4 and at room temp. in hydrazine hydrate. The compds. are stable to heating at 470-500°, as shown by thermogravimetric anal. Thermal mech. curves show that the polymers are not decompd. at 300-50°. The x-ray diffraction anal. of the 3 sep. types of polymer show that all the polymers are amorphous.

~0 Citings

104. Various N-substituted phthalamic acids. Effects on root geotropism in germinating seeds of Lens esculenta

By Pagani, G.; Baruffini, A.; Borgna, P.; Caccialanza, G.
From Farmaco, Edizione Scientifica (1968), 23(5), 448-67. Language: Italian, Database: CAPLUS

Phthalimides I are converted to phthalamic acids II. The II are tested for geotropic activity in germinating L. esculenta seeds and compared to N-(α-naphthyl)phthalamic acid and N-(β-naphthyl)phthalamic acid, which are prepd. from N-(α-naphthyl)phthalimide (III) (m. 180-1°) and N-(β-naphthyl)-phthalimide (IV) (m. 216-17°). Thus, a soln. of phthalic anhydride in HOAc is treated with an equimolar amt. NH3 and the mixt. refluxed 1-3 hrs. to give phthalimide, m. 238°; 0.002 mole phthalamide in alc. is treated with 2 ml. N NaOH and the mixt. heated and acidified to give phthalamic acid. Similarly prepd. are the following V and o-(RNHCO)C6H4CO2H (VI) compds. (R and m.p. V given): 4,1-ClC10H6, 207-9°; 4,1-BrC10H6, 222-4°; 1,2-O2NC10H6, 203-5°; 4,1-O2NC10H6, 221-3°; 2,1-HOC10H6, 221-2°; 5,6,7,8-tetrahydro-1-naphthyl, 146-8°; 1-C10H7NH, 215-16°; Et, 76-8°; Pr, 65-7°; Bu, 33-4°; amyl, 16-17°; isoamyl, 13°; hexyl, 37-8°; cyclohexyl, 168-70°; PhCH2, 115-16°; p-ClC6H4CH2, 123-4°; PhCH2CH2, 130-2°; 2-pyridyl, 227-9°; 3-picolyl, 156-7°; 2-pyrimidyl, 118-20°; 8-quinolyl, 227-9°; 2-benzothiazolyl, 248-9°; 1-phenyl-2,3-dimethyl-3-pyrayolin-5-on-4-yl, 207-9°; 1-phenyl-4-pyrazolyl, 139-41°; and the following I and II (R, R1, R2, R3, R4, and m.p. I given): H, H, H, H, H, 205-7°; Me, H, H, H, H, 180-2°; H, Me, H, H, H, 170-2°; H, H, Me, H, H, 201-2°; Et, H, H, H, H, 137°; H, H, Et, H, H, 175-7°; H, H, F, H, H, 180-2°; Cl, H, H, H, H, 140-2°; H, Cl, H, H, H, 163-4°; H, H, Cl, H, H, 194-6°; H, H, Br, H, H, 204-6°; NO2, H, H, H, H, 202-3°; H, NO2, N, H, H, 245-6°; H, H, NO2, H, H, 270-1°; NH2, H, H, H, H, 190-1°; H, NH2, H, H, H, 189-90°; H, H, NH2, H, H, 249-50°; H, H, NMe2, H, H, 258-60°; AcNH, H, H, H, H, 200-2°; H, AcNH, H, H, H, 216-18°; H, H, AcNH, H, H, 291°; OH, H, H, H, H, 223°; H, OH, H, H, H, 230-1°; H, H, OH, H, H, 295-6°; MeO, H, H, H, H, 157-9°; H, MeO, H, H, H, 126-8°; H, H, MeO, H, H, 160-2°; EtO, H, H, H, H, 122-3°; H, EtO, H, H, H, 116-17°; H, H, EtO, H, H, 204-6°; CO2H, H, H, H, H, 216-17°; H, CO2H, H, H, H, 280-2°; H, H, CO2H, H, H, 288-90°; H, H, SO2NH2, H, H, 333-5°; H, H, Ac, H, H, 240-1°; H, H, Ph, H, H, 290-2°; Me, H, H, Me, H, 160-2°; Me, H, H, H, Me, 203-4°; Cl, Cl, H, H, H, 190-2°; Cl, H, Cl, H, H, 154-5°; Cl, H, H, Cl, H, 206-7°; H, Cl, Cl, H, H, 195-6°; Me, Cl, H, H, H, 215-16°; Me, H, H, Cl, H, 174-5°; H, Cl, Me, H, H, 164-5°; Me, H, NO2, H, H, 200-1°; Me, H, H, NO2, H, 232-4°; NO2, H, Me, H, H, 185-6°; Me, H, NH2, H, H, 182-3°; Me, H, H, NH2, H, 169-70°; NH2, H, Me, H, H, 173-4°; H, NO2, Cl, H, H, 212-13°; H, NO2, H, H, Cl, 195-7°; H, NH2, Cl, H, H, 180-1°; Me, H, Me, H, Me, 173-5°; H, CF3, H, H, H, 112-14°; H, H, Pr, H, H, 145-6°; H, H, Bu, H, H, 138-9°; F, H, H, H, H, 184-6°; H, F, H, H, H, 200-1°; Br, H, H, H, H, 127-8°; H, Br, H, H, H, 164-5°; H, H, BuO, H, H, 146-7°; H, H, PhO, H, H, 160-1°; H, H, MeS, H, H, 202-3°; PhCH2S, H, H, H, H, 114-15°; H, H, PhCH2S, H, H, 261-2°; H, H, SO2CH2CH2OH, H, H, 164-6°; Me, Me, H, H, H, 172-4°; Me, H, Me, H, H, 202-3°; H, Me, Me, H, H, 193-4°; Cl, H, H, H, Cl, 172-4°; H, Cl, H, Cl, H, 203-4°; MeO, H, MeO, H, H, 233-4°; MeO, H, H, Me, H, 230-1°; H, MeO, MeO, H, H, 204-5°; H, EtO, EtO, H, H, 170-1°; Me, H, Cl, H, H, 136-7°; Cl, H, Me, H, H, 176-8°; Cl, H, H, H, Me, 133-4°; Me, H, AcNH, H, H, 224-5°; Me, H, MeO, H, H, 158-9°; MeO, H, H, Me, H, 197-8°; H, CF3, Cl, H, H, 166-8°; H, CF3, NO2, H, H, 150-2°; Cl, H, NO2, H, H, 182-4°; H, NO2, H, CO2H, H, 278-80°; Cl, H, NH2, H, H, 208-10°; Cl, H, H, NH2, H, 182-3°; Cl, H, Cl, Cl, H, 204-5°; H, Cl, OH, Cl, H, 271-3°; MeO, H, MeO, Cl, H, 205-7°; and MeO, H, Cl, MeO, H, 281-2°. The following II [(R, R1, R2, R3, and R4 given): Me, Me, H, H, H; H, Me, Me, H, H; Cl, Cl, H, H, H; Me, Cl, H, H, H; Cl, H, Me, H, H; VI (R = 4,1-BrC10H6), and VI (R = 8-quinolyl)], at a limiting concn. of 10-6M, show geotropic activities similar to those of the standards. Geotropic activity of the II decreases when R - R4 are CO2H, OH, NO2, NH2, NHAc, and hydroxyphenyl; the activity of the II remains unchanged or is increased when R - R4 are halogens or alkyl or alkoxy groups. The activity of the II is decreased when at least one of R and R4 is not H; steric effects are discussed. III and IV are also prepd. from phthalie anhydride.

~11 Citings

105. Fate of thalidomide-14C in the pregnant hamster

By Hague, D. E.; Fabro, Sergio; Smith, Robert Leslie
From Journal of Pharmacy and Pharmacology (1967), 19(9), 603-7. Language: English, Database: CAPLUS, DOI:10.1111/j.2042-7158.1967.tb09596.x

The embryotoxicity and fate of thalidomide-14C (I) in the pregnant European golden hamster were investigated. Daily administration of I (1 or 2 g./kg. orally) to pregnant hamsters on days 4-12 inclusive of pregnancy was not embryotoxic. I (150 mg./kg.) administered at the 204th hr. of pregnancy is well absorbed and about 84% of the 14C is excreted in the urine and 9% in the feces in the 3 days after dosing. The urinary 14C consists of I (3% of dose), α-(o-carboxybenzamido)glutarimide (26%), 2- and 4-phthalimidoglutaramic acids (8%), 2 phthalimidoglutaric acid (0.2%), and 2- and 4-(o-carboxybenzamido)glutaramic acids plus 2-(o-carboxybenzamido)glutaric acid (27%). 14C is present in the embryo, and the relative concns. of radioactivity in the embryo and plasma are about the same at 4, 12, and 24 hrs. after dosing. At 4 hrs. after dosing the embryo contains mainly I, but at 12 hrs. this has largely disappeared and the 14C consists of 7 hydrolysis products. The lack of embryotoxicity of I in the hamster is thus not due to an inability of the teratogen to penetrate to the conceptus.

~0 Citings

106. Fate of thalidomide-14C in the pregnant rabbit

By Fabro, Sergio; Smith, Robert Leslie; Williams, Richard Tecwyn
From Biochemical Journal (1967), 104, 565-9. Language: English, Database: CAPLUS, DOI:10.1042/bj1040565

The fate of thalidomide-14C orally administered to pregnant rabbits at the beginning of the sensitive phase of pregnancy was studied. After the oral administration of thalidomide-14C on the 192nd hr. of pregnancy, ~68% of the radioactivity appears in the urine and 22% in the feces. The urinary 14C is made up as follows (percent of dose): thalidomide (2); α(o-carboxybenzamido)glutarimide (16); 2- and 4-phthalimidoglutaramic acids (11); 2-phthalimidoglutaric acid (0.2); 2- and 4-(o-carboxybenzamido)glutaramic acids and 2-(o-carboxybenzamido)glutaric acid (29). The plasma 14C concn. is maximal at 12 hrs. after dosing, and the radioactivity persists for >58 hrs. At 4 hrs., the main compd. in the plasma is thalidomide, but its concn. steadily declines while the concn. of its hydrolysis products increases. At 12, 24, and 58 hrs. after dosing, radioactivity is present in the embryo and in the maternal tissues examd. The 14C concn. in the embryo is at nearly all times higher than that in the plasma, brain, skeletal muscle, and fat, but lower than that in the liver and kidney. At 4 hrs. after dosing the mother on the 10th day of pregnancy, the specific activities of the embryo and the yolk-sac fluid are similar. Thalidomide is found in the embryo together with 7 of its hydrolysis products for >24 hrs. after dosing. The accumulation of radioactivity in the embryo is due to retention of the polar hydrolysis products.

~6 Citings

107. The fate of thalidomide-14C in the pregnant hamster

By Fabro, Sergio; Hague, D. E.; Smith, Robert Leslie
From Biochemical Journal (1967), 103(1), 26P-27P. Language: English, Database: CAPLUS

Following the daily oral administration of thalidomide (I) in water to hamsters 4-12 days pregnant, no toxicity to embryos was seen at doses of 0.15, 1.0 and 2.0 g./kg. Twenty-four hrs. after the oral administration of 14C-labeled I (0.15 g./kg.) at the 204th hr. of pregnancy, 77% of the dose of 14C was excreted in the urine, while during the next 48 hrs., 7% more was excreted. The urinary 14C-labeled compds. consisted of I (3%), α(o-carboxybenzamido)glutarimide (II) (26%), 2-phthalimidoglutaramic acid (III) plus 4-phthalimidoglutaramic acid (IV) (8%), 2-phthalimidoglutaric acid (0.2%), and 2-(o-carboxybenzamido)-glutaramic acid (V) plus 4-(o-carboxybenzamido)glutaramic acid (VI) plus 2-(o-carboxybenzamido)glutaric acid (VII) (27%). At 4 and 12 hrs., resp., after dosing the mother, the embryos contained I (19.1 and 4.4 g./g.), II (15 and 7.2), III plus IV (0.1 and 2.5), V plus VI plus VII (0.1 and 10.8). Therefore, thalidomide readily penetrated the embryo and persisted in it for more than 12 hrs., suggesting that the lack of toxicity to the hamster embryo was not due to placental impermeability to I.

~0 Citings

108. Preparation of o-nitrophthalic anhydride and its use in the characterization of alcohols and amines

By Chienen Y, Alejandro
From Rev. Fac. Farm. Bioquim., Univ. Nacl. Mayor San Marcos (Lima) (1964), 26(196), 83-107. Language: Spanish, Database: CAPLUS

o-Nitrophthalic acid (I) (26%) was prepd. by nitration of phthalic acid. I was converted to the anhydride (II) by reflux with Ac_2O. II and the appropriate alc. was heated on a steam bath 30 min., the mixt. cooled, and H_2O added. When the b.p. of the alcohol was >150°, pyridine was added as solvent. The product obtained was identified by its phys. properties. The reaction with amines was carried out at ≤145°. Primary amines gave o-nitrophthalamic acids which were dehydrated by heating at 145° to the phthalimides.

~0 Citings

109. Poly(benzoylenebenzimidazoles)

By Colson, J. G.; Michel, R. J.; Paufler, R. M.
From Journal of Polymer Science, Part A-1: Polymer Chemistry (1966), 4(1), 59-70. Language: English, Database: CAPLUS, DOI:10.1002/pol.1966.150040104

Model compds. and poly(benzoylenebenzimidazoles) were prepd., identified, and their properties explored. The reaction of o-$C_6H_4(NH_2)_2$ (I) and phthalic anhydride (II) in $CHCl_3$ or $AcNMe_2$ gives N-(o-aminophenyl)phthalamic acid, a 10% soln. of which (in $AcNMe_2$) on heating at 140° for 30 min. yields N-(o-aminophenyl)phthalamide (m. 194-5°). No benzimidazolecarboxylic acid was detected as a product of this reaction. The imide forms 1,2-benzoylenebenzimidazole (IV) (m. 214-15°) in boiling $PhNO_2$ after 2 hrs. IV can also be prepd. directly from I and II when they are heated in boiling $PhNO_2$ for 90 min. Another model compd. was prepd. from I and pyromellitic dianhydride (V), which gives a mixt. of isomers VIA and VIB. The isomers on heating at 240° and recrystn. from $HCONMe_2$ give the cis- and trans-bislactams VIIA and VIIB. Heating V at lower temps. also gives VI, but crystn. of the product is not successful. The title polymers (VIII) were prepd. from precisely equimolar amts. of 3,3'-diaminobenzidine and IV in II. The intrinsic viscosity of the polymers is 1.0-1.6. The film formed after evapn. of the solvent is converted to the polyimidepolyamine (IX), which forms VIII at 225-50°. Most of the conversion can be accomplished at 200°, but it is not complete until the higher temp. is used. The deep-red film is flexible and has a tensile modulus of 700,000 psi., a tenacity of 11,000 psi., and an elongation of 2%. Differential thermal analysis shows no exotherm <600°. Thermogravimetric analysis in dry air shows no significant wt. loss until 550-600°. Condensation of the very O-sensitive 1,2,4,5-$C_6H_2(NH_2)_4$ and V yields only a polymer of low intrinsic viscosity (0.2-0.3).

~5 Citings

110. The anionic polymerization of allyl acrylate and allyl methacrylate

By Bywater, S.; Black, P. E.; Wiles, D. M.
From Canadian Journal of Chemistry (1966), 44(6), 695-702. Language: English, Database: CAPLUS, DOI:10.1139/v66-097

The low-temp. polymerization of allyl acrylate in PhMe soln. was investigated with BuLi and 1,1-diphenyl-n-hexyllithium as initiators. The latter was also used in a study of the polymerization of allyl methacrylate under the same conditions. The reactions between initiator and monomer were rapid in all cases. More than half of the initiator mols. reacted with monomer acrylic double bonds to start polymer chains, only a few of which grew to a high mol. wt., highly isotactic product. Most of the chains remained as low-mol.-wt., precipitant-sol. product. The remaining initiator mols. reacted with the carbonyl groups of the monomers to produce species that could be detected as allyl alc., after termination of the reaction with HOAc. The allyl double bonds were not involved in reactions during polymerization, but were presumably responsible for the cross-linking that occurred when the polymers were exposed to air.

~4 Citings

111. Reduction of toxicity of radiomimetic alkylating agents in rats by thiol pretreatment. V. Effect of thiol pretreatment on the antitumor action of Merophan

By Connors, T. A.; Jenny, A., Jr.; Whisson, M. E.
From Biochemical Pharmacology (1965), 14(11), 1681-3. Language: English, Database: CAPLUS, DOI:10.1016/0006-2952(65)90024-9

cf. CA 62, 15274e. Neither pretreatment with cysteine or AET protected the host more than the tumor from the toxic action of o-[bis(2-chloroethyl)amino]-DL-phenylalanine (I) in rats with Walker carcinoma; while in mice with ADJ/PC5 plasma tumors, pretreatment with AET enhanced the selective I protection. The pretreatment time was crit. and was max. ~30 min. prior to the intraperitoneal I. The clin. advantage of thiol pretreatment is dubious, since the gain in selective protection is small for the high thiol dose levels required for host protection.

~0 Citings

112. The persistence of maternally administered thalidomide-14C in the rabbit embryo

By Fabro, S.; Smith, R. L.; Williams, R. T.
From Biochemical Journal (1965), 97(2), 14¡Ç½. Language: English, Database: CAPLUS

The measurement of thalidomide-^{14}C 12-58 hrs. after it was given in the 192nd hr. of pregnancy demonstrated that thalidomide, mainly in the form of its metabolites, penetrates and persists in the rabbit embryo and maternal tissues during the entire period of embryonic tetratogenic sensitivity (192nd to 250th hr. of gestation).

~0 Citings

113. The metabolism of thalidomide. Some biological effects of thalidomide and its metabolites

By Fabro, S.; Schumacher, H.; Smith, R. L.; Stagg, R. B. L.; Williams, R. T.
From British Journal of Pharmacology and Chemotherapy (1965), 25(2), 352-62. Language: English, Database: CAPLUS, DOI:10.1111/j.1476-5381.1965.tb02055.x

cf. preceding abstrs. Thalidomide gives rise in the body to 12 hydrolysis products. Since the activity of the parent drug may be mediated by 1 or more of these metabolites the biol. activity of some of these compds. was investigated. A no. of the hydrolysis products were evaluated in biol. tests for embryotoxic activity in rabbits, central nervous depressant activity in rats, and effects on the glutamate decarboxylase, glutamate dehydrogenase, and glutamine synthetase of rat brain. For evaluation of embryotoxic activity the compd. being tested was administered orally or parenterally to pregnant rabbits, usually on days 7-15 of pregnancy. On the 28th day the rabbit was killed; the uterus was examd. for resorption sites, and viable fetuses were examd. for malformations. The central nervous depressant activity of the metabolites was evaluated by observation of changes in the overt behavior of rats after the administration of the compds. and by changes in hexobarbitone-induced hypnosis. Rat brain was a source of the enzymes, glutamate decarboxylase, glutamate dehydrogenase, and glutamine synthetase. Glutamate decarboxylase activity was followed manometrically; glutamate dehydrogenase and glutamine synthetase activities were measured spectrophotometrically. In contrast to thalidomide, none of the hydrolysis products appeared to be significantly embryotoxic in rabbits. None of the metabolites, except α-aminoglutarimide, produced symptoms of overt depression, and they had no effect on the hexobarbitone-induced sleeping time in rats. α-Aminoglutarimide was active in these tests which suggests that the hypnotic activity of thalidomide is related to this particular ring structure. A no. of the hydrolysis products were moderately active as inhibitors of some enzymes concerned with glutamate metab. Glutamine synthetase was inhibited by 2-(o-carboxybenzamido)-glutaric acid and to a lesser extent by 4-(o-carboxybenzamido)-glutaramic acid, and glutamate dehydrogenase was weakly inhibited by these 2 compds. and by phthalic acid, but was inhibited more effectively by DL-glutamic acid. 2-Phthalimido-glutaric acid was a competitive inhibitor of glutamic decarboxylase.

~20 Citings

114. The metabolism of thalidomide. The fate of thalidomide and some of its hydrolysis products in various species

By Schumacher, H.; Smith, R. L.; Williams, R. T.
From British Journal of Pharmacology and Chemotherapy (1965), 25(2), 338-51. Language: English, Database: CAPLUS, DOI:10.1111/j.1476-5381.1965.tb02054.x

cf. preceding abstr. The metabolism of thalidomide in the rabbit, rat, mouse, and guinea pig was investigated. The metabolites of thalidomide present in the urine, blood, and tissues of various species dosed with the drug were characterized by comparing their chromatographic mobility and color reactions with those given by authentic samples of the compds. The rabbit urinary metabolites were isolated in cryst. form by solvent extn. and adsorption chromatography, and their identity was established by analysis, m.p. behavior, and comparison of their ir spectra with those of the authentic compds. When thalidomide is fed to rabbits, rats, mice, and guinea pigs a no. of hydrolysis products appear in the urine. These hydrolysis products are formed by the spontaneous hydrolysis of thalidomide. In addn. the urine of rabbits dosed with thalidomide contains derivs. of 3- and 4-hydroxyphthalic acid; these minor metabolites were not identified. The hydrolysis products appear to be derived by spontaneous breakdown of thalidomide in the body, although it is possible that any of the hydrolytic reactions of thalidomide may be assisted by hydrolases in the body. In rats, some breakdown of thalidomide occurs in the gut before absorption; hydrolysis products are present in the gastrointestinal tract following the oral administration of the drug. Thalidomide and some of its hydrolysis products can be detected in the plasma and brain of rats dosed orally with thalidomide.

~61 Citings

115. The metabolism of thalidomide. The spontaneous hydrolysis of thalidomide in solution

By Schumacher, H.; Smith, R. L.; Williams, R. T.
From British Journal of Pharmacology and Chemotherapy (1965), 25(2), 324-37. Language: English, Database: CAPLUS, DOI:10.1111/j.1476-5381.1965.tb02053.x

When thalidomide is administered to lab. animals, there are excreted in the urine small amts. of unchanged compd. and some 12 substances which are derived from the parent drug by simple hydrolysis. This suggests that thalidomide may be inherently unstable and may undergo spontaneous hydrolysis in the body giving rise to all 12 of its possible hydrolysis products. The spontaneous hydrolysis of thalidomide at various pH values was followed spectrophotometrically and the hydrolysis products formed were identified by their R_f values and color reactions on paper chromatograms. The amt. of each individual hydrolysis product formed in buffers at pH 6, 7.4, and 8.0 was estd. using thalidomide-^{14}C. The hydrolysis products present in an incubated soln. of thalidomide-^{14}C were resolved on a 2-dimensional chromatogram and the radioactivity assocd. with each compd. was estd. using a scintillation spectrometer. At pH values above 6, thalidomide undergoes spontaneous hydrolysis; the rate at which this occurs accelerates with increase in pH. At pH 7, 7.4, and 8 thalidomide has a half-life of 11, 5, and 1.25 hrs., resp. All the substituted amide bonds of the thalidomide mol. are sensitive to hydrolysis, and at pH 7.4, all 12 possible hydrolysis products are formed by splitting of these groups. At all pH values used, the main hydrolysis product is α-(o-carboxybenzamido)glutarimide, but at pH values above 7, increasing amts. of 2- and 4-phthalimidoglutaramic acids are formed. These 3 primary hydrolysis products are also unstable and can undergo further secondary, tertiary, and quaternary hydrolyses. The various substituted amide bonds possess different sensitivities to hydroxyl ion-catalyzed hydrolysis. From pH 6 to 7, only the phthalimide ring undergoes cleavage, and at pH 7 and above, the glutarimide moiety also undergoes hydrolytic splitting.

~87 Citings

116. Intracellular localization of quinidine

By Arora, R. B.; Sharma, J. N.; Tarak, T. K.; Saxena, Y. R.
From Biochemical Pharmacology (1965), 14(11), 1491-8. Language: English, Database: CAPLUS, DOI:10.1016/0006-2952(65)90002-X

The distribution of quinidine in the heart is relatively higher in the ventricles than in the auricles. The intracellular localization of the drug in the left ventricular tissue reveals that the nuclear fraction binds the max., the supernatant fraction being the next, while the sarcosomal fraction shows insignificant affinity. Liver tissue, in contrast, shows that the supernatant fraction retains the highest concn., whereas the nuclear and mitochondrial fractions share about equal amts. The sepn. of the microsomal fraction has not been accomplished. Physicochem. studies demonstrate that the binding of quinidine to the nuclear fraction is rather a phys. process. Quinidine, in the supernatant fractions of both heart and liver, has been shown to be assocd. with the cytoplasmic proteins.

~0 Citings

117. Determination of polymers by gas-liquid chromatography, using an internal standard

By Gratsianskaya, L.P.; Lishtvanova, L. N.; Goryaev, M. I.
From Izvestiya Akademii Nauk Kazakhskoi SSR, Seriya Khimicheskaya (1965), 15(1), 86-8. Language: Russian, Database: CAPLUS

Gas-liq. chromatog. with an internal std. was used to det. the highboiling part of the essential oils and polymers formed by catalytic isomerization of pinene and camphene. Cineole and menthol were used as internal stds. Results are compared with data obtained by the fractional distn. at reduced pressure.

~0 Citings

118. Combined effects of ultra-high pressure and radiation on organic monomers and polymers

By Prince, Martin
From United States Atomic Energy Commission [Unclassified and Declassified Reports Published by the Atomic Energy Commission and Its Contractors] (1964), NYO-3185-1, 82 pp.. Language: English, Database: CAPLUS

Equipment is described in which pressures up to 500,000 psi. were routinely employed. Samples were irradiated with a 1.5-Mev. Dynamitron accelerator; the samples were contained in polyethylene bags and passed through the radiation chamber on a conveyor belt. Acrylamide was irradiated at dose rates of 0.2 to 1.4 × 10^6 rep./hr. and then cooled to -78°. Subsequent polymerization at pressures up to 500,000 psi. indicates that, at any radiation dosage employed, the mol. wt. of polymer obtained from pressurized samples was much higher than that of unpressurized samples. Extent of polymerization was detd. by Br titrn. of unchanged monomer and viscosity measurements. Acrylamide was irradiated with γ-rays from a ^{60}Co source, at dose rates from 0.02 to 3.1 megarep./hr., then stored at -78° in a vacuum. There are great similarities between γ and electron-beam radiation. Higher dose rates of ionizing radiation yield higher polymer conversion. The effect of high pressure on alkyl nitriles was investigated, with polyethylene capsules designed to hold the liquid samples. At 75,000 to 100,000 psi. and 110-50° tetracyanoethylene(TCNE) reacted with an excess of H_2O to yield 2,3,3-tricyanoacrylamide. Reaction of TCNE with MeOH at 75,000 psi. and 130° gave a compd. tentatively identified as Me tricyanoacrylimidate, $(NC)_2C:C(CN)C(:NH)OMe$. Acrylonitrile (I) was irradiated with a high-intensity Co source and polymerized upon irradiation. Reaction with H_2O at 75,000-500,000 psi. and 85 to 200° produced facile hydration to pure polyacrylamlde. Reaction of irradiated I and MeOH at 100,000 psi. produced a polymer with a proposed structure II. Irradiated I reacted with morpholine at 100,000 psi. for 2 hrs. at 130° to yield impure poly(acrylmorpholinoamidine), III. The reactions of pyromellitic dianhydride (IV) with diamines under high pressure were investigated. Reaction of o-phenylenediamine (V) with IV at 89,000 psi. for 16 hrs. at 200° in a 1:1 mole ratio gave a yellow-green solid, which charred at 242° postulated as the amide, VI. Reaction of IV with m-phenylenediamine under the same conditions gave a red-brown material, softening at 250° with the proposed structure VII. Reaction of IV and V in the molar ratio 1:2 gave a yellow material with the proposed structure VIII. Tetraphenylethylene, triphenylethylene, tetrachloroethylene, tetrabromoethylene (IX), tetraiodoethylene, and TCNE could not be polymerized. Only IX and TCNE reacted, with approx. 20% conversion of monomer, possibly yielding a trimer.

~0 Citings

119. The effect of thalidomide and its derivatives on thyroxine-induced metamorphosis of tadpole

By Kim, H. C.; Paik, W. K.; D'Iorio, A.
From Canadian Journal of Biochemistry and Physiology (1965), 43(6), 769-79. Language: English, Database: CAPLUS

Thalidomide and its derivs. are inhibitory to both morphol. and biochem. changes occurring during thyroxine-accelerated metamorphosis of the tadpole. Thyroxine treatment increased the amt. of liver glucosamine, while simultaneous treatment with thalidomide had no effect on the content of this substance. Treatment of tadpoles with thyroxine alone or thyroxine and thalidomide did not affect the adrenaline or noradrenaline contents of kidney and brain. Thus, it appears that the effect of thalidomide on the metamorphosis of the tadpole induced by thyroxine is quite selective. The changes noted were brought about at thalidomide concns. as low as 1 × 10^{-4}M.

~0 Citings

120. Free amino acids in the egg of Ciona intestinalis during some development stages

By Ferrini, U.; Marcante, M. L.; Caputo, A.; Minafra, S.; Reverberi, G.
From Ricerca Scientifica, Parte 2: Rendiconti, Sezione B: Biologica (1964), 5(3), 213-20. Language: English, Database: CAPLUS

The pool of free amino acids was assayed in C. intestinalis eggs from 30 min. after fertilization through the 2nd blastomers, gastrula, and tailbud stages. No quant. or qual. changes were found. Cysteine is either absent or present in very small amts. Tryptophan is absent. Taurine was present in large amts. (2.90 micromoles/mg. total N). 26 references.

~0 Citings

121. Recent advances in the study of the metabolism of toxic substances

By Williams, R. T.
From Annales de Biologie Clinique (1965), 23(1-2), 7-24. Language: French, Database: CAPLUS

The metabolism of several toxic substances specially thalidomide, biphenyl, and arylthioureas was presented. Teratogenicity of thalidomide may be caused by the phthalimide structure N-substituted with a hexagonal ring. Biphenyl is enzymically hydroxylated in positions 2 and (or) 4 depending on species, race, age, and on the presence of other substances. Monoarylthioureas (I) are more toxic than diarylthioureas (II) and have different modes of metabolism. II are hydroxylated and excreted in urine and I are desulfated and metabolized to nontoxic metabolites; H_2S is responsible for the I toxicity. A review with 15 references.

~0 Citings

122. Notes on the relation between the therapeutic and anti-complementary effects of heparin in acquired hemolytic anemia

By Roth, Karl L.
From Annals of Allergy (1965), 23(2), 83-92. Language: English, Database: CAPLUS

Normal subjects were each given subcutaneous injections of 5000-15,000 units of heparin Na. Complement activity decreased only slightly and returned to normal levels within 2 hrs. The level of heparin in the circulating blood had no relation to its ability to prevent blood clotting. Clotting inhibition occurred 120 min. after all traces of heparin had disappeared from the blood. Red blood cells coated with warm autoantibodies gave a neg. Coombs test. Sensitized red blood cells were incubated in heparinized saline and Coombs serum was added. These red blood cells were dissocd. from the adsorbed antibodies after incubation in heparin. Heparin did not dissoc. incomplete cold autoantibodies from red blood cells. The therapeutic effect of heparin in cases of acquired hemolytic anemia where complement may cause red blood cell destruction was not due to inhibition of complement.

~0 Citings

123. Chemistry of N-diacylaminoimidates. IV. Reactions of phthalimidoacetimidates with basic primary amines

By Vargha, Eugen; Balazs, Ileana; Hamburg, Erica
From Studia Universitatis Babes-Bolyai, Chemia (1963), 8(1), 321-9. Language: Romanian, Database: CAPLUS

The reactions of several primary amines with phthalimidoacetimidates in ether were studied. $AmNH_2$ (I), $PhCH_2NH_2$ (II), and cyclohexylamine (III) react like NH_3 to give IV. Thus, 2.68 g. Et phthalimidoacetimidate-HCl in 10 cc. Et_2O was treated with 2.2 cc. II in 20 cc. Et_2O and the mixt. kept 48 hrs. to give 93% IV (R = $PhCH_2$), m. 129-30° ($MeOH$-Et_2OH). Similarly were prepd. 80% IV (R = Am), m. 158.5-9.0°, and IV (R = cyclohexyl), 90% m. 173-6° (Me_2CO). IV (R = cyclohexyl) was treated 8 hrs. at 20° with 10 cc. EtOH-HCl to give 80% V (R = cyclohexyl), m. 247-8° (Me_2CO-Et_2O). Similarly were prepd. 85% V (R = Am), m. 191-3° (EtOH-HCl) and 90% V (R = $PhCH_2$), m. 179-80° (decompn.) ($MeOH$-Et_2O). With equimolar quantities of K_2CO_3, IV gave VI. Thus, 1 g. IV (R = Am) in 10 cc. hot H_2O was treated with a satd. soln. of K_2CO_3 until neutral to give 95% VI (R = Am), m. 205-6° (EtOH-H_2O). Also prepd. were 70% VI (R = $PhCH_2$), m. 207-8°, and 40% VI (R = cyclohexyl), m. 225° (EtOH-H_2O). With dil. HNO_3 IV (R = Am) gave a nitrate, without hydrolysis of the amido group, m. 173-4°. Refluxing VI with a satd. alc. soln. of picric acid gave the following VII picrates. (R, % yield, and m.p. given): Am, 60, 140° (decompn.); $PhCH_2$, 90, 186-7°; cyclohexyl, 98, 162-3°. The structures of IV-VII were verified by their chem. properties and ir spectra.

~0 Citings

124. N-Diacylaminoiminoesters. III. Phthalimidoacetamidines

By Vargha, E.; Balazs, I.; Balog, A.
From Studia Universitatis Babes-Bolyai, Chemia (1963), 8(1), 311-19. Language: Romanian, Database: CAPLUS

cf. CA 58, 4421h. The conversion of I (R = Me or Et) into amidines was studied. Thus, I was treated with 10% NH_3 in abs. EtOH at 0° to give 93% 2-$H_2NCOC_6H_4CONHCH_2C(NH_2)$:NH.HCl II, m. 187-8°. II with 5% HCl in abs. EtOH at 80° gave quant. III (R = H), m. 256°, but with 2N HCl 2-$HO_2CC_6H_4CONHCH_2C(NH_2)$:NH.HCl (IV) resulted (m. 241-2°). With $PhNH_2$ I gave 86% III (R = Ph) (V), m. 238-9°. V with N NaOH gave 2-$^-O_2CC_6H_4CONHCH_2C(NHR)$:$N^+H_2$ (VI) (R = Ph, VII) which affords V by boiling with 10% HCl in abs. EtOH. With picric acid in EtOH VII gives the picrate of V. When III was treated with dil. NaOH in an equimol. amt., 90% VI (R = H), m. 235-6°, was obtained. The structures of these amidines were verified by their ir spectra.

~0 Citings

125. Thalidomide metabolism. II. Mechanism of the action of thalidomide

By Fabro, S.; Schumacher, H.; Smith, R. L.; Williams, R. T.
From Bollettino - Societa Italiana di Biologia Sperimentale (1963), 39(24), 1925-9. Language: Unavailable, Database: CAPLUS

The effects of I and III on Streptococcus faecalis (folic acid dependent) and Lactobacillus leichmanii (vitamin B_{12} dependent) were examd. No growth inhibition was obsd. The effects in vitro of I and its metabolites on the glutamine synthetase (XI), L-glutamic decarboxylase (XII), and glutamic dehydrogenase (XIII) activity of the rat brain were tested. 4-(o-Carboxybenzamido)glutaramic acid inhibits II and XIII slightly; V inhibits XII, and 2-(o-carboxybenzamido)glutaric acid inhibits all 3 enzymes. I and other metabolites have no action. The inhibitory activity is obtained at concns. of 10^{-3}-10^{-2}M. The metabolites of I, when they are administered orally to rabbits, possess no teratogenic activity. The acidic and polar properties of I metabolites prevent them from penetrating into cells and thus exerting their teratogenic activity. 25 refs.

~0 Citings

126. Thalidomide metabolism. I. Spontaneous thalidomide hydrolysis

By Fabro, S.; Schumacher, H.; Smith, R. L.; Williams, R. T.
From Bollettino - Societa Italiana di Biologia Sperimentale (1963), 39(24), 1921-5. Language: Unavailable, Database: CAPLUS

Spontaneous hydrolysis of thalidomide (I) in vitro in 0.1M pH 7.4 phosphate buffer at 37° in 24 hrs. gave 12 compds.: 4-phthalimidoglutaramic acid (II), 2-phthalimidoglutaramic acid (III), α-(o-carboxybenzamido)glutarimide (IV), 2-phthalimidoglutaric acid (V), 4-(o-carboxybenzamido)glutaramic acid, 2-(p-carboxybenzamido)glutaramic acid, 2-(o-carboxybenzamido)glutaric acid, phthalic acid (VI), α-aminoglutarimide (VII), isoglutamine (VIII), glutamine (IX), glutamic acid (X). Hydrolysis products were isolated by 2-dimensional paper chromatography with 7:7:6 C_5H_5N-AmOH-H_2O for the 1st direction and H_2O-satd. 10:1 BuOH-HOAc for the 2nd direction. After incubation for 1 hr. at 18° and pH 7.4, I is 4-5% hydrolyzed; at 37° this increases to 8%. After 24 hrs. at 18° the hydrolysis is 50%, whereas at 37° it is 80%. The hydrolysis increases with pH values: it is 77% at pH 7.8 after 5 hrs., and at pH 8.8 it is complete after 1 hr. In the spontaneous hydrolysis of I at pH 7.4, after 1 hr. II, III, IV, and VII are present; after 4 hrs. in addn. to these compds. V, VI, VIII, IX, and X appear. After 24 hrs. all 12 products are present. By hydrolyzing in vitro at pH 6-6.8, the phthalimide ring is labile, whereas that of glutarimide is stable; at pH 6.8-7.4 the glutarimide ring is also rapidly hydrolyzed. In rabbit, guinea pig, and mouse urine, which has a pH > 7, there is more II in comparison with other hydrolysis products, whereas in rat urine, which has a pH < 7, IV is the main metabolic product. By alkalizing rat urine by oral administration of 100 mg. NaOAc 30 min. before treatment with I, the major metabolite is II.

~1 Citing

127. Interactions between various diuretics

By Sala, G.
From Atti della Accademia Medica Lombarda (1963), 18, 1029-41. Language: Unavailable, Database: CAPLUS

A review.

~0 Citings

128. Antimitotic effect of thalidomide and its metabolites on chick embryo blood cells

By Villa, L.; Valentini, R.; Taglioretti, D.; Eridani, S.
From Haematol. Latina (Milan) (1963), 6(3), 217-21. Language: English, Database: CAPLUS

cf. Lancet 1963-I,725. Inhibitory action of thalidomide (I) on development of chick embryos is proportional to dose and is most evident when I is subjected during the earliest stages of development. Growth defects and anatomic abnormalities are observed most frequently in embryos of eggs injected with I at 24 hrs. and incubated for long periods. Chromosome fragmentation and conglutination are observed at all stages of cell division in contrast to effects of colchicine and vinblastine which affect a critical stage of cell division. Reinoculation of blood from embryos previously injected with I into other eggs also results in antimitotic effects but requires higher doses. Doses of 2 mg. of metabolites of I (Faigle, et al., CA 57, 15741b) depress mitotic activity to a greater extent than does I. With higher doses, a complete disappearance of initial mitotic figures in the prophase stage occurs. An inhibitory effect on metabolism of glutamic acid by I and its metabolites is the probable mechanism.

~1 Citing

129. Inhibitory activity of benzoyl hydrazides and hydrazine on the growth of influenza virus in chick embryo lung tissue culture

By Kundin, W. D.; Robbins, Mary Louise; Smith, P. K.
From Experientia (1964), 20(8), 438-9. Language: English, Database: CAPLUS

The hydrazides of anthranilic acid, benzoic acid, 2-methoxybenzoic acid, m-nitrobenzoic acid, and salicylic acid, as well as hydrazine itself, were found to inhibit the growth of influenza virus in tissue culture. Anthranilic acid hydrazide (I) inhibited virus growth when added within 6, but not 8, hrs. after virus inoculation. After a 30-hr. incubation there was a greater than 10,000-fold difference between the tube receiving only virus and the tube receiving I and virus simultaneously. I had no significant virucidal activity. Neither anthranilic acid, quinic acid, kynurenic acid, tyrosine, phenylalanine, p-aminobenzoic acid, aniline, tryptophan, indole, nor nicotinic acid reversed the antiviral activity, suggesting that the inhibitory property may be assocd. with the hydrazide moiety. I (4 mg.) inoculated into the allantois of each of 6 fertile hen eggs failed to inhibit the growth of influenza virus. I was nontoxic to mice at a concn. of 0.5% in the diet.

~2 Citings

130. Quantitative paper-chromatographic determination of some glycoalkaloids

By Pierzchalski, T.
From Tagungsber. Deut. Akad. Landwirtschaftswiss. Berlin (1961), No. 27, 171-6. Language: Unavailable, Database: CAPLUS

Grind leaves contg. 30-50 mg. alkaloid glycosides with sand and shake 2 hrs. with twice the amt. of HOAc. Filter, then ext. for 1 hr. with half the amt. of 2% HOAc. Make combined exts. alk. with NH_4OH and heat to 70-80°. After 1 hr., filter off the crystals, wash 2-3 times with 1% NH_4OH, dry at 60°. Reflux the dried crystals 2-3 hrs. with 150 ml. EtOH, filter, evap. to dryness on a water bath, and dissolve the residue in 6 ml. of 2% HOAc. For the paper-chromatographic sepn. of the alkaloid glycosides, immerse Whatman No. 1 paper strips in pH 4.5 buffer and air-dry. Spot the alkaloid soln. on the paper and develop at 20° either by the ascending or descending techniques. For the former, a suitable system is BuOH-H_2O-satd. Me_2CO-H_2O (3 : 5 : 1); for the latter, the system BuOH-H_2O-satd. Me_2CO-H_2O (3 : 4 : 1) is suitable. The spots are located by use of Dragendorff reagent and are eluted with 5 ml. of 2% HOAc for 24 hrs. at room temp. For color development, add 4 ml. concd. H_2SO_4 dropwise over 4 min. to 2 ml. of eluate, with cooling, then add 3 ml. of fresh 1% CH_2O soln. within 2 min. Let stand 90 min. and det. the absorbance using a yellow filter.

~0 Citings

131. Paper chromatography of thalidomide and its hydrolytic products

By Schumacher, H.; Smith, R. L.; Stagg, R. B. L.; Williams, R. T.
From Pharmaceutica Acta Helvetiae (1964), 39(6), 394-8. Language: German, Database: CAPLUS

By the use of 1- and 2-dimensional paper chromatography with mobile phase 1 consisting of pyridine-AmOH-H_2O (7:7:6) and mobile phase 2 consisting of BuOH-AcOH (100:10), H_2O satd., thalidomide and 12 hydrolytic products were sepd. R_f values and identification reactions are given.

~0 Citings

132. Biological activity of thalidomide metabolites

By Fabro, S.; Schumacher, H.; Smith, R. L.; Stagg, B. L.; Williams, R. T.
From Biochemical Journal (1964), 90(1), 5¡Ç½-6¡Ç½. Language: Unavailable, Database: CAPLUS

2- and 4-Phthalimidoglutaramic acid, 2- and 4-(o-carboxybenzamido)glutaric acid, phthalic acid, and α-aminoglutarimide were administered orally or i.p. (300-400 mg./kg.) to rats 30 min. before injection i.p. of hexobarbital-Na (70 mg./kg.). Only the last compd. extended the sleeping time as does thalidomide. None of the compds. except thalidomide was toxic to fetuses of rabbits. 2-(o-Carboxybenzamido)glutaric acid only inhibited glutamate synthetase, glutamate dehydrogenase, and glutamate decarboxylase (50% at 1 mM). Thalidomide had no effect on O uptake by homogenates of chinchilla fetuses, indicating that the compd. may not affect energy-yielding processes.

~0 Citings

133. Distribution in organs and elimination of 9-[(N-methyl-14C-3-piperidyl)methyl]thioxanthene hydrochloride (methixene)

By Aebi, H.; Lauber, E.; Lehner, H.; Michaelis, W.
From Arzneimittel-Forschung (1964), 5, 92-5. Language: Unavailable, Database: CAPLUS

After intraperitoneal and intravenous (i.v.) injection of a single dose (10 mg./kg.) of the title compd. (methixene, Tremaril) (I) into mice, the amt. of activity in blood, liver, and the gastrointestinal tract was independent of the mode of administration, but is higher in the carcasses and the central nervous system after i.v. injection. The elimination of $^{14}CO_2$ in the expired air, a consequence of N-demethylation, sets in immediately after administration and is finished after 12 hrs. The excretion of labeled compds. in feces and urine starts 2-8 hrs. after application and continues for about 24 hrs. Within 48 hrs. after injection 32% of the activity administered is found in the expired air, about 30% in feces, and about 15% in urine. The chem. nature of the labeled compds. found in the liver, the gastrointestinal tract, and brain after i.v. injection has been detd. by paper chromatography. Five min. after administration unchanged I and its 2 isomeric sulfoxides (II) were present in all organs tested. I remained longer in the organs than II. As the only probable transmethylation product, choline-^{14}C, has been detd. in the gastrointestinal tract 12 hrs. after administration. 18 references.

~0 Citings

134. Imidazole dyes. XI. Condensation of pyromellitic acid with o-phenylenediamine

By Arient, J.; Havlickova, L.
From Collection of Czechoslovak Chemical Communications (1963), 28, 2534-6. Language: German, Database: CAPLUS

A mixt. of 1 mole pyromellitic acid (I) (m. 272°), 1.2 moles o-$(H_2N)_2C_6H_4$ (II), and AcOH was refluxed 3 hrs., the alkali-sol. green-yellow product extd. first with boiling AcOH to remove the starting compds., and then with EtOH to remove III, R_f 0.75; repptn. of the EtOH-insol. green-yellow product from concd. H_2SO_4 gave IV, m. 346-51°, R_f 0.85 in a 1:2 C_5H_5N-H_2O mixt. satd. with 1-$C_{10}H_7Br$ (V) on Whatman No. 1 paper impregnated with V. A refluxing soln. of 2 moles II in C_5H_5N was treated with a soln. of I tetrachloride in C_5H_5N, the mixt. refluxed 5 hrs., the yellow ppt. collected, refluxed with Ac_2O 3 hrs., the soln. clarified hot, allowed to cool, the cryst. product collected and sepd. by crystn. from Ac_2O into orange VI, m. 398-400° ($HCONMe_2$, $PhNO_2$) (blue vat), and yellow 4,5-bis(2-benzimidazolyl)phthalic anhydride.

~1 Citing

135. Thalidomide and its antifolic acid activity

By Lammers, W.; Manten, A.
From Pharmaceutisch Weekblad (1963), 98, 458-60. Language: Unavailable, Database: CAPLUS

The antifolic acid activity of thalidomide and 7 of its metabolites (Faigle, et al. CA 57, 15741b) was tested on Streptococcus faecalis. No inhibitory action could be demonstrated.

~0 Citings

136. Biochemical distinction between nonsteroid antiinflammatory and analgesic drugs

By Whitehouse, M. W.
From Journal of Pharmacy and Pharmacology (1963), 15(8), 556-7. Language: Unavailable, Database: CAPLUS

As alternatives to steroids, phenylbutazone (Butazolidine) (I) and 2-$AcOC_6H_4CO_2H$ (II) are prescribed extensively for the management of rheumatic diseases. I and II are moderately potent analgesics and exhibit antipyretic activity. These facts have lent credence to a belief, that antiinflammatory activity among nonsteroid drugs is either synonymous with or largely overlaps analgesic activity in nonnarcotic drugs. This belief has been strengthened by recent reports that mefenamic acid (III) possesses antipyretic, antinociceptive, and antiinflammatory activity in animals (Winder, et al., CA 58, 7272g) and is a promising analgesic for clin. use. Adams (CA 54, 17695g) suggested that it was not justifiable to classify all analgesic-antipyretic drugs as a single group and presented data that nonnarcotic analgesics could be divided into 2 groups: (1) those including 2-$HOC_6H_4CO_2H$ (IV), II, and I, which suppressed inflammation in lab. animals, and (2) other analgesics, including several derivs. of IV, which were inactive in an antiinflammatory test (UV light-induced erythema). This subdivision of the nonnarcotic analgesics into at least 2 groups, according to their antiinflammatory properties, is supported by biochem. data. Oxidative phosphorylation in isolated liver mitochondria is uncoupled by nonsteroid antiinflammatory drugs and certain antiinflammatory steroids. These compds. also uncouple oxidative phosphorylation in extrahepatic tissues, notably in connective tissues. The following correlation was made of the antiinflammatory activity of some non-narcotic analgesics with their effects upon (a) oxidative phosphorylation in respiring liver mitochondria with succinate as substrate and (b) phosphate metab. in a connective tissue (cartilage) (detd. in vitro according to Whitehouse and Haslam, CA 58, 7262h) [analgesic, antiinflammatory activity, effect on oxidative phosphorylation (liver), effect on phosphate metab. (cartilage) given[(+ indicates activity and - indicates no activity): I, +, +, +; oxyphenbutazone, +, +, +; amidopyrine (V), +, -, -; antipyrine, -, -, -; paracetamol, -, -, -; PhNHAc -, -, -, p-$EtOC_6H_4NHAc$, -, -, -, IV, +, +, +; 2-$HOC_6H_4CONH_2$, -, -, -; 2- and 4-hydroxyisophthalic acids, -, -, -; III, +, +, +; 3-hydroxycinchophen, +, +, +. V was the only compd. which failed to uncouple oxidative phosphorylation in vitro but has some antiinflammatory activity in vivo. Two of its metabolites, 4-aminoantipyrine and 4-(N-acetamido)pyrine, were as inactive in vitro as V. Other analgesics that failed to uncouple oxidative phosphorylation at a concn. of 2 m-mol were: salicyloylpiperidine, phenylamidol-HCl, morphine sulfate, pethidine-HCl, and carisoprodol; none of these exhibits significant antiinflammatory activity in lab. animals. Na aurothiomalate and Au Na thiosulfate, which are not analgesics, uncouple oxidative phosphorylation in liver mitochondria at concns. of 3 and 0.3 m-mol, resp. Their Au-less analogs had no effect on oxidative phosphorylation at 5 m-mol concn. Antimalarial aminoquinolines, such as chloroquine and hydroxychloroquine, which manifest antirheumatic activity only after prolonged administration, could be distinguished biochem. from other nonsteroid antiinflammatory drugs by their failure to uncouple oxidative phosphorylation at concns. up to 5 m-mol, even after preincubation with liver mitochondria for 4 h. at 2°.

~1 Citing

137. Imidazole dyes. VIII. Reaction of substituted phthalic anhydrides with 1,2-diaminobenzene

By Arient, J.; Havlickova, L.
From Collection of Czechoslovak Chemical Communications (1963), 28, 1885-94. Language: German, Database: CAPLUS

cf. CA 59, 4070f. A series of dyes contg. an imidazole ring was prepd. by condensation of substituted phthalic anhydrides with o-$C_6H_4(NH_2)_2$ (I) and subsequent transformation. Thus, refluxing a soln. of 5 g. 3-nitrophthalic anhydride (II) in 100 ml. MeOH contg. 2.3 ml. 36% HCl with a soln. of 2.8 g. I in 10 ml. MeOH 3 hrs. gave 2'-amino-2-carboxy-6-nitrobenzanilide, m. 187° (dil. AcOH). Refluxing 5 g. II with 2.8 g. I in 100 ml. AcOH 3 hrs. gave 1.5 g. (20.3%) 2-(6-nitro-2-carboxyphenyl)benzimidazole (III), m. 256° ($HCONMe_2$), which (12 g.) refluxed with 100 ml. Ac_2O gave 9 g. (80.3%) IV (X = 6-NO_2) m. 241° (Ac_2O). Adding 5 g. II in 30 ml. AcOH over 30 min. to a boiling soln. of 5.2 g. I in 50 ml. AcOH, refluxing the mixt. 3 hrs., sepg. from the cooled mixt. 0.7 g. (9.4%) III, and adding H_2O pptd. 6.2 g. (66.8%) 3-nitro-1,2-bis(2-benzimidazolyl)benzene (V), m. 345-6° ($HCONMe_2$). Redn. of 15 g. III in 350 ml. HCl with 69 g. Sn at reflux temp. (4 hrs.) gave 7 g. (52.2%) 2-(6-amino-2-carboxyphenyl)benzimidazole, m. 210-20°, which refluxed 4 hrs. with Ac_2O gave, by extn. of the solid product with PhMe, 16.1% IV (X = 6-NHAc), m. 253.5-4°, and 13.6% 2-(6-acetamido-2-carboxyphenyl)benzimidazole, m. 272.5-3° (C_5H_5N). Redn. of 5 g. V with 20 g. Sn in 100 ml. HCl by refluxing 3 hrs. afforded 4 g. (87.3%) 3-amino-1,2-bis(2-benzimidazolyl)benzene, m. 376-8°, which was acetylated (Ac_2O, reflux 4 hrs.) to yield 3-acetamido-1,2-bis(2-benzimidazolyl)benzene, m. 247° (C_5H_5N). Refluxing 5 g. 4-nitrophthalic anhydride (VI) with 2.8 g. I in 100 ml. AcOH 3 hrs. gave 0.62 g. (23.3%) 4-nitro-1,2-bis(2-benzoimidazolyl)benzene, m. 332-3.5° (C_5H_5N), and 0.31 g. (14.5%) 2-(4-nitro-2-carboxyphenyl)benzimidazole, (VII), m. 298-9°. Refluxing 12 g. VII with 100 ml. Ac_2O 3 hrs. gave 9.5 g. (84.5%) IV (X = 4-NO_2), m. 248-9° (Ac_2O), also obtained by sublimation of VII at 220° and 1 mm. Boiling 12 g. VII in 300 ml. concd. HCl with 55 g. Sn 5 hrs. afforded 3.2 g. (29.7%) 2-(4-amino-2-carboxyphenyl)benzimidazole (VIII), m. 220°, which (2.3 g.) heated with 30 ml. Ac_2O gave 1.26 g. (47%) IV (X = 4-NHAc), m. 327-8° ($HCONMe_2$), also obtained by subliming VIII at 220° and 1 mm. to give IV (X = 4-NH_2), m. 202-3°, and acetylating this compd. Refluxing 5 g. VI with 2.8 g. I in 100 ml. MeOH gave 7.2 g. (92.3%) 2'-amino-4-nitro-2-carboxybenzanilide, m. 128° (H_2O). Redn. of 15 g. 4-nitro-1,2-bis(2-benzimidazolyl)benzene with 80 g. Sn in 350 ml. concd. HCl afforded 7.2 g. (66%) 4-amino-1,2-bis(2-benzimidazolyl)benzene, m. 280300°, which, when treated with Ac_2O, gave 69.5% 4-acetamido1,2-bis(2-benzimidazolyl)benzene, m. 297-8° ($HCONMe_2$). Refluxing 5 g. 3-acetamidophthalic anhydride with 2.6 g. I in 100 ml. AcOH 3 hrs. yielded 1.2 g. (16.9%) 3-acetamido-N-(2-aminophenyl)phthalimide (IX), m. 268° ($HCONMe_2$), and by pptn. of the mother liquors with H_2O, 1.8 g. of a product whose extn. with PhMe afforded 0.51 g. (7.2%) IV (X = 3-NHAc), m. 239.5-40° (PhMe), and 0.7 g. (10.5%) 2-(3-acetamido-2-carboxyphenyl)benzimidazole (X), m. 264-5° ($HCONMe_2$). Hydrolysis of IX with cold concd. HCl yielded a compd., probably 3-amino-N-(2-aminophenyl)phthalimide, m. 243°. Refluxing 0.15 g. X in 20 ml. of a soln. of 1 g. NaOH in 10 ml. H_2O and 10 ml. MeOH for 2 hrs., neutralizing the mixt. with HCl, and evapg. to dryness gave 2-(3-amino-2-carboxyphenyl)benzimidazole, m. >300°, which was sublimed at 220° and 1 mm., to give IV (X = 3-NH_2), m. 198-9°. Refluxing 5 g. 4-acetamidophthalic anhydride with 2.6 g. I in 100 ml. MeOH yielded 0.16 g. (2.2%) 4-acetamido-N-(2-aminophenyl)phthalimide, m. 314-15°, and 0.05 g. of a compd. which, when refluxed with Ac_2O, gave a compd. probably 2-(5-amino-2-carboxyphenyl)benzimidazole, m. 253-4° (Ac_2O). Adding over 2 hrs. 8.6 g. 2,5-$Me(O_2N)C_6H_3COCl$ in 300 ml. C_5H_5N to a boiling soln. of 4.5 g. I in 100 ml. C_{HN} gave 3.43 g. (33.1%) 2-(2-methyl-5-nitrophenyl)benzimidazole, m. 200-0.5° (EtOH), which was refluxed with a mixt. of $K_2Cr_2O_7$, H_2O, H_2SO_4, and AcOH to give 58.8% 2-(5-nitro-2-carboxyphenyl)benzimidazole, m. 260-2°; sublimation at 200-10° and 5 mm. afforded IV (X = 5-NO_2), m. 235-6°.

~2 Citings

138. Past, present, and future of metalliferous dyes

By Wahl, Henri
From Teintex (1963), 28, 257-69. Language: Unavailable, Database: CAPLUS

Review with 8 references.

~0 Citings

139. The use of cyanide-form ion exchange resins in the preparation of nitriles

By Gordon, M.; Griffin, C. E.
From Chemistry & Industry (London, United Kingdom) (1962), 1019-20. Language: Unavailable, Database: CAPLUS

Reaction of o-phthalimidomethylbenzyl bromide with NaCN in EtOH gave a complex product instead of the nitrile; solvolytic cleavage of the phthalimido group and CN^- catalyzed condensation was indicated. To develop an alternative method of synthesis, a soln. of $PhCH_2Br$ in 95% EtOH was stirred at 65° with 1 equiv. Amberlite IRA-400 (CN form) (I) to a neg. Br^- test (1.5 hrs.). After filtration, 53% $PhCH_2CN$, b_{15} 110-11% was obtained. Similarly p-bromo- (II) and p-methylbenzyl bromides and CH_2:$CHCH_2Br$ gave 98, 43, and 23% yields of the corresponding nitriles. II with I in Et_2O, tetrahydrofuran, C_6H_6, and $HCONMe_2$ gave approx. 20% nitrile in all cases. The reaction is believed to be the 1st noted involving the breaking and formation of covalent bonds on the surface of I with the resin anion becoming covalently bound in the product. p-$O_2NC_6H_4CH_2Br$ with I gave 23% 1,2,3-tris(p-nitrophenyl)-2-cyanopropane, m. 203-4% hydrolysis of which gave the corresponding acid, m. 223-5°. Neither 4,4'-dinitrostilbene nor p-$O_2NC_6H_4CH_2CN$ were detected in the reaction products.

~2 Citings

140. Chelate forming α-aminothiocarboxylamides

By Jenni, J.; Kuhne, H.; Prijs, B.
From Helvetica Chimica Acta (1962), 45, 1163-71. Language: German, Database: CAPLUS

Some potential chelating agents, thioamides of α-amino acids, were synthesized from α-aminonitriles and H_2S. DL-α-Aminopropionitrile (I) (decompd. at 125-8°, 14% yield), DL-α-aminobutyronitrile (II) (m. 135-8°, 26% yield), DL-α-aminovaleronitrile (III) (m. 148-50°, 13% yield), and DL-α-aminocapronitrile (IV) (no m.p., 18% yield) were synthesized from NH_4Cl, NaCN, and aldehydes. The following α-hydroxynitriles were obtained also by distg. the ether filtrates after pptg. the HCl salts of I-IV: DL-α-hydroxypropionitrile (20% yield, b_{12} 85-6°, n 1.400, d. 0.993), DL-α-hydroxybutyronitrile (30% yield, b_{112} 93°, n 1.412, d. 0.961), I) DL-α-hydroxycapronitrile (22% yield, b_{12} 113-14°, n 1.4128, d. 0.924), and DL-α-hydroxyvaleronitrile (18% yield, b_{12} 101°-3, n 1.420, d. 0.936). α-Hydroxycaproamide (10% yield) was also a by-product of the synthesis of IV. DL-α-Phthalimidopropionitrile (V) (13.5% yield, m. 138 9°), DL-α-phthalimidobutyronitrile (VI) (20% yield, m. 95-6°), DL-α-phthalimidovaleronitrile (VIII) (18% yield, m. 85-6°), and DL-α-phthalimidocapronitrile (VIII) (5.5% yield, 46-50°) were made by the Gabriel synthesis from K phthalimide and α-brmnonitriles in dimethylformamide. DL-α-Phthalimidopropionamide (IX) (81% yield, m. 209-11°) and DL-α-phthalimidocaproamide (X) (80% yield, m. 203-5°) were made by successive reactions of DL-alanine and DL-norleucine with phthalic anhydride, $SOCl_2$, and NH_3. Heating X at 200-3° 20 min. gave DL-α-phthalimidocaproamide (m. 172-3°, 90% yield). DL-α-Phthalimidopropionic acid was esterified to Me DL-α-phthalimidopropionate (XI) (m. 27-9°, 90% yield) with MeOH and dry HCl. Methanolic NH_3 at 0° converted the ester to Me DL-α-phthalamidopropionate (XII) (70% yield, m. 156-8°). Heating XII at 180-90° converted it back to XI (94% yield). XI in ethanolic NH_3 at room temp. yielded IX (34% yield, m. 170-1°), which was heated at 210-20° to obtain DL-α-phthalimidopropionamide (80% yield, m. 188-208°). This was converted to V with P_2O_5. The analogous Et ester reactions gave a better yield (92%) of IX. $CaCl_2$-dried H_2S was slowly bubbled 1 week through a soln. of 200 ml. abs. C_6H_6, 5 ml. Et_3N, and 3.85 g. V to obtain 4.25 g. DL-α-phthalimidothiopropionamide (XIII) (m. 164-8°). DL-α-Phthalimidothiobutyramide (95% yield, m. 198-201°), DL-α-phthalimidothiovaleramide (XV) (73% yield, m. 148-50°), and DL-α-phthalimidothiocaproamide (XVI) (83% yield, m. 134-7°) were prepd. similarly from the resp. nitriles, as were DL-α-aminothiobutyramide (48% yield, m. 108-9°) and DL-α-aminothiovaleramide (XVII) (60% yield, m. 108-9°). XV (3 g.) was vigorously stirred into 50 ml. 2N NaOH and then neutralized with 10% HCl. DL-α-(o-Carboxybenzamido)thiovaleramide (XVIII) (94% yield, m. 178-80°) pptd. VIII (3 g.) was refluxed 10 min. in 30 ml. 10% HCl, then kept 12 hrs. at 0°. Phthalic acid (94%.) was filtered off. The filtrate was concd. under vacuum at room temp., redissolved in the min. amt. H_2O, neutralized with $KHCO_3$, and kept at 0°. XVII pptd. in 69% yield. DL-α-(o-Carboxybenzamido)thiocaproamide (95% yield, m. 183-4°) and DL-α-aminothiocaproamide (38% yield, m. 92-5°) were prepd. similarly. Although DL-α-(o-carboxybenzamido)thiopropionamide (97% yield, m. 182-6°) was prepd, similarly from IX, no DL-α-aminothiopropionamide was obtained.

~3 Citings

141. Intermolecular dehydrogenation with ultraviolet light. VI. Photoreactions of various aliphatic amines

By Pfordte, Klaus; Leuschner, Gerhard
From Justus Liebigs Annalen der Chemie (1961), 646, 25-30. Language: Unavailable, Database: CAPLUS

cf. CA 55, 23450f; 56, 7119f. Irradiation of various primary, secondary, and tertiary amines for 24 hrs. with unfiltered ultraviolet light from a high-pressure Hg arc gave rise to dehydrogenation at the C atom linked to the amine. Irradiation of $PrNH_2$ for 24 hrs. gave a 5% mixt. of 3,4-diaminohexanes; the racemic form (I) b. 100°, $n^{20}{}_D$ 1.4161; colorless mobile liquid, turning green in air [monopicrate, m. 138-9° ($CHCl_3$)]; and the meso form (II) b. 180°, $n^{20}{}_D$ 1.4621, viscous yellow liquid darkening in air, reacting with picric acid to form a resin. Both I and II reacted with HIO_4 in aq. AcOH at 20° giving EtCHO (dinitrophenylhydrazone, m. 152°). With phthalic anhydride (III) in Me_2CO, I gave $HO_2CC_6H_4CONHCH(Et)CH(Et)NH_2$, m. 141.5°. I formed no complex with $Cu(OH)_2$. II gave N with HNO_2 and formed a resin with III: II formed a bright blue complex with $Cu(OH)_2$. On irradiation, $BuNH_2$ gave 7% of a mixt. of dl-4,5-diaminooctane (IV) b. 138-40°, $n^{20}{}_D$ 1.4230 and the meso isomer (V) b. 240-45°, $n^{20}{}_D$ 1.4630. IV and V react with HIO_4, giving PrCHO (2,4-dinitrophenylhydrazone, m. 122-4°). IV reacted with III, giving the acid phthalamido deriv., m. 144.5 (Me_2CO). V (but not IV) gave a blue complex with $Cu(OH)_2$. V gave no identifiable deriv. with picric acid or with III. iso-$BuNH_2$ on irradiation formed the dl-3,4-diamino-2,5-dimethylhexane (VI), b. 127°, $n^{20}{}_D$ 1.4530 (picrate, m. 152°) and the meso isomer (VII), b. 220°, $n^{20}{}_D$ 1.4542. Both VI and VII react with HIO_4, giving iso-PrCHO. VII but not VI gave a blue complex with $Cu(OH)_2$. VI but not VII reacted with III, giving the acid phthalamido deriv., m. 133° (Me_2CO). Et_2NH on irradiation gave largely dl-2,3-bis-ethylaminobutane, pale yellow, b. 165°, $n^{20}{}_D$ 1.4461, which with HNO_2 gave a yellow bis(nitrosoamine), m. 77° (aq. EtOH). VIII forms a monopicrate, m. 153° ($CHCl_3$-petr. ether). In this reaction, only very small amts. of the meso isomer of VIII, b. 240°, were formed. Both VIII and its isomer gave AcH with HIO_4. Irradiated Et_3N gave dl-2,3-bis(diethylamino)-butane, b. 135, $n^{20}{}_D$ 1.4310; picrate, m. 177° (PhMe) and the meso isomer, b. 208° $n^{20}{}_D$ 1.4409; impure HCl salt m. approx. 216° (decompn.). Neither of these isomers gave complexes with $Cu(OH)_2$; neither of them reacted with HIO_4.

~0 Citings

142. Derivatives of glycineamidine

By Bose, Ajay K.; Greer, Francis; Gots, Joseph S.; Price, Charles C.
From Journal of Organic Chemistry (1959), 24, 1309-13. Language: Unavailable, Database: CAPLUS, DOI:10.1021/jo01091a038

cf. CA 53, 16008i. Phthalimidoacetonitrile (I) was converted through the imidoester hydrochloride, o-$C_6H_4(CO)_2NCH_2C(OEt):NH.HCl$ (II) to various derivs. of glycineamidine. I and o-$C_6H_4(CO)_2NCH_2CO_2Et$ (III) were treated with NH_3 under various conditions. $H_2C:NCH_2CN$ (1.7 g.) and 3.7 g. o-$C_6H_4(CO)_2O$ in 20 ml. C_5H_5N heated 1 hr. on a steam bath and the pale yellow soln. poured onto 150 g. cracked ice, the product washed with H_2O, and dried yielded 25.5% I, m. 129.5-30.5°, together with 1.08 g. o-$C_6H_4(CO_2H)_2$, obtained by acidifying the filtrate. I (25 g.) in 75 ml. dry dioxane and 6.5 g. alc. bubbled through 20 min. at 0° with dry HCl (15 g.) and the mixt. kept 16 hrs. at 0-5° yielded 89% II, m. 258-60° (decompn.), similarly prepd. from I in 85.5% yield by heating $NCCH_2NH_2.0.5H_2SO_4$ with o-$C_6H_4(CO)_2O$ in C_5H_5N at 80-90°. II (15 g.) in 50 ml. alc. kept 2 days at 0° with 3 g. NH_3 in 40 ml. alc. yielded 95.5% o-$H_2NOCC_6H_4CONHCH_2C(NH_2):NH.HCl$ (IV), m. 175-8° (decompn.). II in H_2O within 1 min. gave III, m. 109-11°, also obtained in 85 per cent yield by heating II in H_2O and cooling. II kept molten 2-3 min. gave o-$C_6H_4(CO)_2NCH_2CONH_2$, λ 2.82, 2.92, 3.03, 5.62, 5.82, 5.95, 6.20 μ, also obtained directly from I by reaction with concd. H_2SO_4 or a basic soln. of H_2O_2, in accordance with the imino ester structure for II. IV (2 g.) shaken a few min. with H_2O and the alc.-washed product dried 3.5 hrs. at 110° in vacuo gave the salt-like o-$^-O_2CC_6H_4CONHCH_2C(NH):NH_2\ ^+.H_2O$ (V), m. 218-19° (decompn.), converted by HBrAcOH to o-$C_6H_4(CO)_2NCH_2C(NH_2):NH.HBr$ (VI). IV (5.5 g.) stirred 16 hrs. at 20° with 100 ml. alc. contg. 1.8 g. Na and 3.2 g. $H_2C(CO_2Et)_2$ and the solvent evapd., the residue taken up in H_2O, and the soln. acidified with HCl gave 0.5 g. phthalimide, m. 224-5°. IV (2 g.) shaken occasionally during 1 hr. in 15 ml. 2:1 AcOH-48% HBr and kept overnight before filtering gave 0.7 g. VI, m. 262-4° (decompn.), λ 2.9, 3.13, 5.65, 5.9, 6.0 μ. Addn. of Et_2O to the filtrate gave 0.55 g. VI (total yield 56%). V (0.9 g.) added to 10 ml. 3:2 AcOH-48% HBr and the soln. kept 3 hrs., the product washed with Et_2O, and dried in vacuo at room temp. gave 0.5 g. o-$HO_2CC_6H_4CONHCH_2(NH_2):NH.HBr$ (VII), m. 261° (decompn.), λ 3.0, 5.80, 5.92, 6.0-6.15 μ. Finely powd. II (1 g.) shaken vigorously with satd. $NaHCO_3$ in CH_2Cl_2 at room temp. and the dried org. layer evapd. gave 0.48 g. III, m. 110-11°, λ 5.82, 5.75, 5.65 μ. II (3 g.) shaken 15 min. at 0-5° in satd. $NaHCO_3$ in CH_2Cl gave 2.2 g. solid (VIII), m. 154-7°, repeatedly recrystd. from Et_2O-$CHCl_3$ or MeOH to give silky needles of o-$C_6H_4(CO)_2NCH_2C(:NH)OEt$ (IX), m. 159-61°, λ 3.0, 5.65, 5.85, 6.0 μ. Chromatography of VIII over Florasil and elution with ligroine (60-5°) and Me_2CO (3-6%) gave III. Further elution with 91:9 ligroine-Me_2CO yielded IX. Finely powd. IX (1 g.) kept 1.5 hrs. in 50 ml. alc. contg. 8% NH_3 and the filtered soln., kept 3 days before evapg. in vacuo below 40° yielded material, m. 218-20°, washed with alc. and Et_2O and dried in vacuo at 50° to give 0.45 g. V, converted by keeping 24 hrs. in 5 ml. 3:2 AcOH-48% HBr to yield 0.33 g. VI. III (2.5 g.) in 15 ml. 58% NH_4OH stirred vigorously 15 min. and kept 30 min. at 20°, dild. with 100 ml. H_2O, and the product washed twice with 50 ml. MeOH and once with 50 ml. Et_2O yielded 1 g. o-$H_2NCOC_6H_4CONHCH_2CONH_2$ (X), m. 263-4°, λ 2.94, 2.97, 3.00, 3.16, 6.02, 6.13, 6.20, 6.40 μ. III (2 g.) in 50 ml. alc. almost satd. with NH_3 at room temp. with occasional stirring and filtered after keeping 3 days gave 1.25 g. X. III (5.8 g.) shaken occasionally in 100 ml. NH_4OH and kept 4 days, the clear soln. evapd. on a steam bath, and the residue crystd. from 95% alc. gave 3.8 g. o-$C_6H_4(CO)_2NCH_2CONH_2$ (XI), m. 253-4°. The mother liquor yielded a 2nd crop of XI, contaminated with X. Use of alc. NH_3 gave only XI. III (1 g.) and 20 ml. NH_4OH kept 4 days and the clear soln. concd. in vacuo at 35-40°, the product (0.3 g.) washed with alc., and dried yielded o-$H_4NO_2CC_6H_4CONHCH_2CO_2NH_4$ (XII), λ 2.89, 2.94, 3.08, 3.16-3.29, 5.94, 6.06, 6.23, 6.30, 6.38-6.48 μ. Powd. I (1.5 g.) stirred 20 min. with 9 ml. 58% NH_4OH, the product washed with small amts. of H_2O and with MeOH, and dried gave 0.6 g. slightly impure o-$H_2NCOC_6H_2CONHCH_2CN$ (XIII), λ 3.02, 3.20, 5.97, 6.03, 6.15, 6.47 μ. Powd. I (5 g.) added to 25 ml. ice-cold alc. satd. with NH_3 and the clear filtered soln. kept 2 hrs., the ppt. washed with Et_2O, and dried gave impure XIII. Powd. I (1 g.) kept overnight in 45 ml. NH_4OH and the filtered soln. kept 2 days at room temp., filtered, and evapd. in vacuo below 40° yielded XII. I (2 g.) and 50 ml. NH_4OH kept 3 days, the clear soln. evapd. on a steam bath, and the residue crystd. from alc. gave 1 g. impure XI. I (0.5 g.) in 3 ml. H_2SO_4 kept overnight at 20° and poured over cracked ice gave quant. XI. I (0.3 g.) kept 3 hrs. in 30 ml. alc., 10 ml. 0.1N NaOH, and 6 ml. 30% H_2O_2 and the mixt. concd. in vacuo gave 0.14 g. 1st crop of XI. XIII (0.4 g.) kept molten 20 min. with evolution of NH_3 and the cooled melt taken up in alc., the hot soln. decolorized with C, and cooled yielded 83% I. XIII (0.5 g.) in 3 ml. concd. H_2SO_4 kept 16 hrs. at 20° and poured onto crushed ice, the solid product washed with H_2O, and dried yielded 28% XI. XIII (0.2 g.) in 30 ml. 2:1 alc.-0.1N NaOH kept 16 hrs. at 20° with 3 ml. 30% H_2O_2 and the clear soln. evapd. in vacuo at 20° gave 0.05 g. o-$C_6H_4(CO)_2NH$, m. 232-4°. XIII (2.5 g.) in 100 ml. dry C_6H_6 and 1 ml. alc. satd. 10 min. with HCl at 0° and the mixt. kept overnight at 0-5°, the product washed with Et_2O and C_6H_6, and the impure I boiled with H_2O gave XI, which with alc. NH_4OH afforded impure XIII. XIII (0.5 g.) in 50 ml. concd. NH_4OH kept 3 days at 20° and the clear soln. evapd. in vacuo at 50° yielded 0.15 g. light brown XII. II showed greater than 50% inhibition of growth of Escherichia coli at 100 γ/ml., whereas V showed less than 50% inhibition at 1000 γ/ml.

~3 Citings

143. Some derivatives of 1-aminocyclopentanecarboxylic acid and related compounds

By Connors, T. A.; Ross, W. C. J.
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Some derivs. of 1-aminocyclopentanecarboxylic acid (I) were prepd. for examn. as tumor growth inhibitors. These included several esters and acyl derivs., and dipeptides derived from glycine and DL-phenylalanine. Substituted derivs. of the parent amino acid included the 2-carboxy-, 3-carboxy-, and the 2,3-dibenzo compds. 1-Aminocyclopropane- (II), -cyclobutane- (III), -cyclohexane- (IV), -cycloheptane- (V), and -cycloöctane + carboxylic acid (VI) were also prepd. and some new derivs. were described. Two isomers of 2-aminocyclopentane-1-carboxylic acid (VII and VIII) were prepd. and characterized. α-Cyclopropyl- (IX) and α,β-diphenylalanine (X) were also obtained. Hydantoin-5-spirocyclopentane (14.9 g.), 53.3 g. $Ba(OH)_2.8H_2O$, and 325 ml. H_2O heated 2 hrs. at 160°, cooled, pptd. $BaCO_3$ removed, 12 g. $(NH_4)_2CO_3$ added, and the filtrate concd. gave 11.7 g. I, prisms, m. 328-9°. I (8 g.) suspended in 200 ml. MeOH satd. with dry HCl, refluxed 3 hrs., evapd., and the oil separated gave I Me ester.HCl, needles, m. 207-8° (decompn.). The I Et ester-HCl obtained as needles, m. 228° (decompn.) (Et_2O-alc.); I isopropyl ester-HCl, needles, m. 180° (Et_2O-Me_2CHOH). The N-acetyl deriv. of I was obtained by heating (2 hrs. at 100°) 10 g. I with 9.5 ml. Ac_2O and 25 ml. AcOH; it formed needles, m. 195-7° (alc.-ligroine). I (2 g.) in 28 ml. 2N Na_2CO_3 treated with 3.2 ml. BzCl gave 1-benzamidocyclopentanecarboxylic acid, m. 214-15° (aq. alc.). I (3 g.) and 4.5 g. phthalic anhydride were heated 1 hr. at 170° in a sealed tube; the MeOH sol. fraction gave 1-phthalimidocyclopentanecarboxylic acid-$2H_2O$ (XI), m. 156-8°. The MeOH insol. portion was probably 3,6-dioxopiperazine-2,5-bis(spirocyclopentane), m. above 360°. XI (1.17 g.) and 0.94 g. dicyclohexylcarbodiimide (XIa) in 10 ml. tetrahydrofuran gave dicyclohexylurea. The next day, 450 mg. of solid was obtained, which afforded the anhydride, prisms, m. 118° (Et_2O-pentane) (when heated 2 hrs. with H_2O it gave XI). XI (9 g.) and 30 ml. $SOCl_2$ contg. 0.5 ml. C_5H_5N was stirred 1 hr. at room temp. and then 0.5 hr. at 40°; after removal of excess $SOCl_2$ under reduced pressure at 40°, the residual acid chloride (XII) was extd. and crystd. to give prisms, m. 44° (pentane). XII shaken with concd. NH_4OH gave 1-phthalimidocyclopentanecarboxamide (XIII), prisms, m. 215° (alc.). An attempt to remove the phthaloyl group from XIII by the action of 1 equiv. of alc.-N_2H_4 led to a sparingly sol. hydrazide, m. 210° (alc.). When this hydrazide was heated with dil. HCl, I was formed. I Et ester-HCl (10 g.) suspended in dry Et_2O, 7.2 ml. NEt_3 added, the mixt. stirred 2 hrs., the filtrate evapd., and the residual oil washed with concd. NH_4OH, stirred 3 days at 30° (sealed flask), and evapd. gave 2.2 g. 1-aminocyclopentanecarboxamide (XIV), m. 95-6° (Me_2CO-pentane). Heating 125 mg. XIV with 125 mg. phthalic anhydride 1 hr. at 90° gave XIII. I (12 g.) and 16.2 g. KOCN in sufficient H_2O for soln. heated 1 hr. and treated with HCl to pH 5 gave 1-ureidocyclopentanecarboxylic acid, small prisms, m. 203° (decompn.). I Et ester-HCl (0.5 g.) in 2 ml. C_5H_5N contg. 0.5 mg. BzCl heated 15 min. at 100°, dild. with H_2O, extd. with Et_2O, washed with 2N HCl, and the product chromatographed on activated Al_2O_3 gave Et 1-benzamidocyclopentanecarboxylate, prismatic needles, m. 76-7° (ligroine). XII (23.5 g.) added during 0.5 hr. to 11.2 g. glycine ester-HCl and 24.8 g. NEt_3 in 80 ml. dioxane at 5-10°, the mixt. stirred 4 hrs., 1.1 l. H_2O added, most of the dioxane distd., and the product extd. gave 16.3 g. N-(ethoxycarbonylmethyl)-1-phthalimidocyclopentanecarboxamide (XV), needles, m. 90° (Et_2O-pentane). Isobutyl chloroformate (1.3 ml.) added slowly to a cooled soln. of 2.6 g. XI and 1.4 ml. NEt_3 in 25 ml. $CHCl_3$, the mixt. stirred 2 hrs. at 5°, 1.4 g. of glycine ester-HCl and 1.4 ml. NEt_3 added in 10 ml. $CHCl_3$ during 0.5 hr., stirred 3 hrs., washed, and evapd. gave XV. 80% $N_2H_4.H_2O$ (0.12 ml.) kept a few min. with 680 mg. of the phthalimido ester in 10 ml. alc. gave the hydrazide (XVI), plates, m. 205° (aq. alc.) (decompn.). XVI was readily sol. in 2N HCl; when this soln. was heated, phthalhydrazide separated; the soln. filtered, the filtrate evapd., and the product crystd. gave 1-amino-N-carboxymethylcyclopentanecarboxamide-HCl (XVII), prisms, m. 200-1° (alc.Et_2O). In a typical expt., 13 g. of the phthalimido ester gave 11.3 g. XVI and 4.5 g. XVII. An aq. soln. of XVII passed slowly through Amberlite IR-4B gave the hemihydrate, plates, m. 269-70° (aq. alc.). In an attempt to obtain the anhyd. peptide, the hemihydrate was sublimed at 180°/0.02 mm.; it gave 3,6-dioxopiperazine-2-spirocyclopentane (XVIII), m. 275-80° (decompn.) (H_2O). Evapn. of a soln. of XVIII in concd. HCl gave XVII. Phthaloylglycyl chloride (8.06 g.) in 35 ml. dioxane slowly added to 7.5 g. I Et ester-HCl in 10.8 ml. NEt_3 and 40 ml. dioxane, the mixt. stirred 3 hrs., H_2O added, the solid collected, and the product crystd. gave 9.1 g. Et 1-phthaloylglycylamidocyclopentanecarboxylate (XIX), m. 146-7° (aq. alc.). Phthaloylglycyl chloride (4.47 g.) in dry dioxane added dropwise during 35 min. to 2.58 g. I, 1.21 g. $Mg(OH)_2$, and 75 ml. H_2O, stirred 20 min. at 5°, the mixt. allowed to reach room temp. during 20 min,. and acidified gave 900 mg. 1-phthaloylglycylamidocyclopentanecarboxylic acid (XX), needles, m. 218-20° (H_2O). XX (1.73 g.) and 0.26 ml. 80% $N_2H_4.H_2O$ in 500 ml. alc. refluxed 2 hrs. and the residue digested 10 min. at 40° with 2N HCl gave XIX. Et phthaloylglycylcyclopentanecarboxylate (40.65 g.) and 7.65 g. $N_2H_4.H_2O$ in 1.5 l. alc. refluxed 3 hrs. and the product isolated as above gave 23.07 g. 1-glycylamidocyclopentanecarboxylic acid, m. 202-4°; HCl salt, needles, m. 216° (Me_2CO). I Et ester-HCl (11.6 g.), 8.28 ml. NEt_3, and 150 ml. tetrahydrofuran stirred 0.5 hr., the NEt_3.HCl collected, the filtrate kept 5 hrs. at room temp. with 17.6 g. N-phthaloyl-DL-phenylalanine, 12.36 g. XIa added, 5.4 g. dicyclohexylurea removed, acid added, the mixt. filtered again, and the filtrate evapd. to dryness gave 17.6 g. Et 1-(N-phthaloyl-DL-phenylalanylamido)cyclopentanecarboxylate(XXI), prismatic needles, m. 142-4° (C_6H_6-pentane). XXI (4.34 g.), 0.65 ml. 80% $N_2H_4.H_2O$, and 50 ml. alc. refluxed 2 hrs., the solid collected, the filtrate evapd., and the residue extd. with 2N HCl gave 1.5 g. 1-(DL-phenylalanylamido)cyclopentanecarboxylic acid-HCl, m. 200° (decompn.) (alc.-Et_2O). The free dipeptide formed needles, m. 268-70° (slight decompn.). DL-Phenylalanine Et ester-HCl (5.16 g.) and 6.3 g. NEt_3 in 50 ml. tetrahydrofuran stirred 0.5 hr., kept overnight with XII (from 5.85 g. acid) in 20 ml. tetrahydrofuran at 0°, H_2O added, the solvent evapd., and the residue extd. with Et_2O gave 8.5 g. of a gum. Also, DL-phenylalanine ester in tetrahydrofuran added to 5.85 g. XI and 4.7 g. XIa in 25 ml. tetrahydrofuran soon gave cyclohexylurea; the whole stirred overnight at room temp. gave 9 g. of a gum, which did not cryst. The product from the above reaction (13.2 g.) refluxed 24 hrs. with 2 ml. $N_2H_4.H_2O$ in 250 ml. alc. gave 5-benzyl-3,6-dioxopiperazine-2-spirocyclopentane, m. 262-4° (MeOH). Et 2-oxocyclopentanecarboxylate (XXII) (5 g.), 3.2 g. NaCN, and 200 ml. $(NH_4)_2CO_3$ soln. in aq. alc. heated 16 hrs. at 58-60°, evapd., and the soln. made acid, extd. with Me_2CO, and chromatographed on Al_2O_3 gave 2 g. hydantoin-5-spirocyclopentane-2'-carboxylic acid Et ester, rosettes, m. 145° (Et_2O-ligroine). XXII (5 g.), 6 g. NH_4Cl, 10 g. $(NH_4)_2CO_3$, 6 g. NaCN, 100 ml. alc., and 80 ml. H_2O heated 26 hrs. at 60-70° in a pressure bottle, the product isolated, refluxed with 150 ml. 4N HCl, and the filtrate evapd. to half vol. and treated with C gave 2.7 g. hydantoin-5-spirocyclopentane-2'-carboxylic acid (XXIII), needles, m. 225-6° (H_2O). XXIII (10.9 g.) and 15.5 g. $Ba(OH)_2.8H_2O$ in 100 ml. H_2O heated 1.5 hrs. at 170-80° in a pressure bottle, 16.3 g. $(NH_4)_2CO_3$ added, the residue obtained (on evapn.) rubbed with MeOH, and the product sepd. gave 1-aminocyclopentane-1,2-dicarboxylic acid (XXIV), m. 223° (decompn.). When MeOH or Me_2CO was added to a concd. soln. of XXIV, NH_4 H 1-aminocyclopentane-1,2-dicarboxylate (XXV) separated, plates, m. 249° (decompn., from 226°). XXV (0.5 g.), 2 ml. Ac_2O, and 50 ml. AcOH refluxed 2 hrs., evapd. to dryness, and the residue

dissolved in Me_2CO and Et_2O added gave the N-acetyl deriv. of XXIV, prisms, m. 226-8°. 3-Oxocyclopentanecarboxylic acid (6.4 g.), 9.8 g. NaCN, and 19.2 g. $(NH_4)_2CO_3$ in 200 ml. 1:1 aq. alc. heated 25 hrs. in a pressure bottle at 65-70° gave 4.7 g. hydantoin-5-spirocyclopentane-3'-carboxylic acid (XXVI), m. 243-5°, apparently a mixt. of isomers. XXVI (5.5 g.) was hydrolyzed to give 3.9 g. of a deliquescent solid, m. 160-85°. On repeated treatment with hot MeOH, this material gave 1-aminocyclopentane-1,3-dicarboxylic acid-H_2O, prisms, m. 264° (H_2O); hemihydrate m. 277°; N-acetyl deriv., prisms, m. 225-6° (Et_2O-alc.). The hydantoin (8.5 g., m. 242-3°) derived from 1-indanone heated 2 hrs. at 180° with 20.8 g. $Ba(OH)_2.8H_2O$ and 150 ml. H_2O gave 1-aminoindan-1-carboxylic acid (XXVII). XXVII (1 g.), 1 ml. Ac_2O, and 20 ml. AcOH refluxed 2 hrs. gave the N-acetyl deriv., needles, m. 259-60° (decompn.) (aq. alc.). Diethyl acetamidomalonate (21.7 g.) dissolved in a soln. of 2.5 g. Na and 200 ml. alc., 25.7 g. Et γ-bromocrotonate added, the mixt. refluxed 5 hrs., most of the solvent removed, H_2O added, and the soln. extd. with Et_2O, washed, and evapd. gave a low-melting solid. This solid (18.1 g.) in 25 ml. alc. shaken over PtO_2 and H during 2 days gave 6.5 g. triethyl 1-acetamidobutane-1,1,4-tricarboxylate (XXVIII), m. 98-9° (Et_2O). XXVIII (4.4 g.) refluxed 3 hrs. with 25 ml. concd. HCl and 25 ml. H_2O and the soln. evapd. to dryness gave 2.4 g. of solid, m. 138-40°, which in 12 ml. H_2O and 15 ml. alc. treated with C_5H_5N gave 1 g. α-aminoadipic acid, m. 199-201° (aq. alc.). Dry NH_3 passed through a mixt. of 20 g. XXII and 11 g. NH_2NO_3 (after 2 days the mass liquified), the whole shaken with Et_2O, and the upper layer concd. gave 13.5 g. Et 2-aminocyclopent-1-enecarboxylate (XXIX), m. 55-7° (Et_2O-pentane). XXIX and excess BzCl kept 1 hr. gave Et 2-benzamidocyclopent-1-enecarboxylate, needles, m. 110-1° (Et_2O-MeOH). XXIX (7.75 g.) in 30 ml. AcOH contg. 200 mg. PtO_2 absorbed 1 mole H during 19 hrs.; the mixt. filtered and evapd., the product refluxed 1 hr. with 25 ml. concd. HCl and 25 ml. H_2O, and the soln. concd. gave a gum, which yielded some NH_4Cl. When the MeOH ext. was kept several days, a solid, m. 210°, separated; heating this hydrochloride gave a sublimate of 1-cyclopentene-1-carboxylic acid (XXX), m. 120°. The mother liquors from the HCl salt dild. with H_2O and passed through Amberlite resin and the absorbed material eluted with dil. NH_4OH and evapd. gave an amino acid, m. 227-35° (decompn.), considered to be bis(2-carboxycyclopentyl)amine. XXX was prepd. essentially by the method of Cook and Linstead (CA 28, 6116[9]). Dehydration of 1-cyano-1-hydroxycyclopentane with $SOCl_2$ in C_5H_5N gave higher yields of 1-cyano-1-cyclopentene. XXX (20 g.) in 250 ml. NH_4OH heated 2 days at 150° in an autoclave, 10 g. basic Cu carbonate added to the cooled mixt., the soln. heated, the soln. evapd., the blue plates collected, the Cu salt purified by redissolving in excess dil. NH_4OH and then boiling off the NH_3 gave a dihydrate; on drying, a monohydrate was obtained. H_2S passed into a soln. of the Cu salt in dil. HCl, the inorg. product removed, and the filtrate evapd. gave VII.HCl, prisms, m. 165-70°. The m.p. was not definite, since decompn. with the formation of XXX occurred at the point of fusion. An aq. soln. of 1 g. VII.HCl digested with 1 g. Ag_2O, AgCl removed, and the soln. evapd. gave VII.H_2O, m. 242-3° (decompn.); VII gave a weak ninhydrin test. Et benzamidocyclopentanecarboxylate formed flattened needles, m. 98-9°. The mother liquors from the sparingly sol. Cu salt were acidified, the unchanged cyclopentanecarboxylic acid was extd. with Et_2O, and the aq. layer satd. with H_2S, filtered, and evapd. Extn. with MeOH left some $(NH_4)_2SO_4$; evapn. with HCl gave 1 g. VIII.HCl, prismatic needles, m. 210-12°; N-benzoyl ethyl ester, needles, m. 91.5-3.0° (Et_2O-ligroine). Diethyl cyclopropane-1,1-dicarboxylate (125 ml., b_{12} 116-19°) and 625 ml. NH_4OH stirred 2 days in a pressure bottle gave 49.9 g. of the diamide, m. 199°. The diamide was converted into 40.1 g. hydantoin-5-spirocyclopropane, m. 220-2°. A mixt. of this hydantoin (10 g.) and 25 g. $Ba(OH)_2.8H_2O$ in 300 ml. H_2O heated 40 min. at 155° in a pressure bottle, and the product isolated gave 4.5 g. II, needles, m. 248-9° (decompn.) (Me_2CO); HCl salt, plates, m. 220-3° (decompn.). Diethyl cyclobutane-1,1-dicarboxylate (84 g.) was converted similarly to the diamide, m. 280-3°, in 16.9 g. yield, and then into 13 g. of the spirohydantoin, m. 224°, which on hydrolysis (as described) gave 8.3 g. III, m. 290° (decompn.) (aq. alc.); HCl salt, plates, m. 260-5°; N-acetyl deriv. m. 179° (Me_2CO-ligroine); N-benzoyl deriv. m. 204-5° (Et_2O). Hydantoin-5-spirocyclohexane, needles, m. 221-5° (alc.), was hydrolyzed by $Ba(OH)_2$ at 160° to give IV, plates, m. 330-40° (H_2O); N-benzoyl deriv. m. 198-200° (aq. alc.). Similarly, hydantoin-5-spiro(3-methylcyclohexane) gave 1-amino-3-methylcyclohexanecarboxylic acid, prisms, m. 305-10° (H_2O); N-benzoyl deriv., plates, m. 220-1° (aq. alc.). The hydantoin from α-tetralone, m. 248° (alc.), gave 1-amino-1,2,3,4-tetrahydro-1-naphthoic acid, prisms, m. 258-60°; N-benzoyl deriv. m. 174-8° (alc.). The hydantoin from β-tetralone m. 268-9° (prisms from aq. alc.). It yielded 2-amino-1,2,3,4-tetrahydro-2-naphthoic acid, m. 297-303° (H_2O); N-benzoyl deriv., prisms, m. 213-15° (alc.). V, prepd. in the usual manner from the spirohydantoin, m. 320° (decompn.) (H_2O); N-benzoyl deriv., needles, m. 202-3° (Et_2O). The appropriate hydantoin, m. 246°, similarly afforded VI, plates, m. 321° (decompn.) (aq. Me_2CO); N-benzoyl deriv., m. 226° (aq. Me_2CO). 5-Methyl-5-propylhydantoin, m. 123-4°, on hydrolysis with $Ba(OH)_2$ afforded α-methylnorvaline, subliming above 312°; benzoyl deriv., prisms, m. 160-1° (Et_2O-ligroine). Hydantoin-5-spiro(2-methylcyclohexane) (10 g., m. 223-5°) in 35 ml. H_2O contg. 15 ml. concd. H_2SO_4 was heated 4 hrs. at 160-80°, 85 ml. $Ba(OH)_2.8H_2O$ added, and the ppt. centrifuged. The filtrate gave 1-amino-2-methylcyclohexane-carboxylic acid, needles, m. 330°; N-benzoyl deriv., prisms, m. 196° (aq. alc.). Cyclopropyl Me ketone (30 g.), 42 g. NaCN, and 165 g. $(NH_4)_2CO_3$ in 200 ml. H_2O and 200 ml. alc. heated 5 hrs. at 58-60°, the soln. kept overnight, evapd., acidified, the ppt. collected, and the liquors evapd. gave 32 g. 5-cyclopropyl-5-methylhydantoin (XXXI), prisms, m. 148-51° (Et_2O). XXXI (13.8 g.), 27 g. Ba-$(OH)_2.8H_2O$, and 200 ml. H_2O heated 1 hr. at 160°, 13.8 g. $(NH_4)_2CO_3$ added, the mixt. filtered, evapd., and the product crystd. gave IX, m. 290-2°; HCl salt, prisms, m. 264-6° (H_2O); HBr salt, prisms, m. 254-7° (H_2O); N-benzoyl-α-cylopropylalanine, plates, m. 195-6° (H_2O). IX (10 g.) in 30 ml. alc. satd. with dry HCl was refluxed 2 hrs.; the next day, evapn. gave 13.3 g. IX.HCl (used without purification). This was stirred 2-3 hrs. in 70 ml. tetrahydrofuran and 9.6 ml. NEt_3; 7.93 g. phthaloylglycine and 8.1 g. XIa in 10 ml. tetrahydrofuran added, the mixt. stirred 16 hrs., and the EtOAc soln. of the residue (obtained after removal of the tetrahydrofuran) washed and recrystd. gave 9.3 g. N-phthaloylglycyl-α-cyclopropylalanine Et ester (XXXII), m. 145-6° (EtOAc-pentane). XXXII (9.51 g.) and 1.75 g. 80% $N_2H_4.H_2O$ in 200 ml. alc. refluxed 2 hrs., the alc. removed, 150 ml. 2N HCl added, the soln. heated 5 min., filtered, evapd., and the mixt. chromatographed gave 2 g. glycyl-α-cyclopropylalanine, m. 251-2° (aq. alc.); HCl salt, prisms, m. 152-3° (aq. alc.). 5-Benzyl-5-phenyl-hydantoin (18 g.), m. 215°, and 34.4 g. $Ba(OH)_2.8H_2O$ in 600 ml. H_2O heated 10 hrs. at 160-80° gave 12 g. X, needles, m. 274-5° (H_2O); N-benzoyl deriv., prisms, m. 84° (aq. alc.); N-carbamoyl deriv., prisms, m. 190-1° (aq. alc.).

~18 Citings

144. Conversion of 3-amino-4-phenylazophenol to benzimidazole derivatives

By Guarneri, Mario
From Annali di Chimica (Rome, Italy) (1957), 47, 163-6. Language: Unavailable, Database: CAPLUS

cf. G. B. Crippa and M. Guarneri, C.A. 47, 9935h. Expts. are described which confirm the structure 3-amino-4-phenylazophenol assigned to the coupling product from m-$H_2NC_6H_4OH$. 3-Phthalimido-4-phenylazophenol (10 g.) in 180 cc. 8% Na_2CO_3 is treated gradually with 17 g. $Na_2S_2O_4$ at 60-70° (the color fades to pale yellow), filtered hot and cooled, the pptd. Na salt filtered off and dissolved immediately in a min. of hot H_2O, and treated with HCl to ppt. N-(2-amino-5-hydroxyphenyl)phthalamic acid hydrochloride (I), gives an intense violet with very dil. $CuSO_4$ and NaOH; when sublimed it evolves HCl and H_2O and forms orange flocks, m. 320°, of benzoylene-3-hydroxybenzimidazole (II). I (5 g.) heated to boiling in 50 cc. H_2O ppts. yellow needles of 3-phthalimido-4-aminophenol, m. 270° (decomp. to II). II (2 g.) in 10 cc. dil. HCl heated to boiling, filtered, and cooled ppts. 6-(2-carboxyphenyl)-3-hydroxybenzimidazole hydrochloride, m. 248° (decomp. to II). I (3 g.) and 3.4 g. o-$C_6H_4(CO)_2O$ heated 30 min. at 170-80° then cooled give 3,4-bis(phthalimido)phenol, m. 326°.

~0 Citings

145. N-Phthalylglutamic acid imide

By Kunz, W.; Keller, H.; Muckter, H.
From Arzneimittel-Forschung (1956), 6, 426-30. Language: Unavailable, Database: CAPLUS

The chem. and pharmacol. properties of a new sedative 3-phthalylamino-2,6-dioxopiperidine (I) are described. I forms white needlelike crystals m. 271°, is sparingly sol. in H_2O, MeOH, EtOH, Me_2CO, AcOEt, AcOBu, and AcOH, very sol. in dioxane, $HCONMe_2$, and pyridine, and insol. in Et_2O, $CHCl_3$, C_6H_6. On account of its low soly. in H_2O and its low toxicity no oral and parenteral $L.D._{50}$ could be detd. in the white mouse; more than 5000 mg./kg. are tolerated s.c., 1500 i.p., and 5000 orally. A dog of 6.5 kg. wt. receiving 10 g. I by stomach tube showed no toxic signs. Feeding of 500 mg./kg. daily for 30 days to mice, 200 to rats and 100 to rabbits and guinea pigs did not produce toxic signs in the animals. For the testing of the sedative action of I a special system of cages was devised permitting simultaneous recording of the motility of a group of mice. The onset of the sedative action of I is rapid and the effect of long duration, the smallest dose inducing sleep is about 2.5 times that of phenylethylbarbituric acid. I does not produce initial excitation of the animal even in high doses. I has no effect on cardiac action, blood pressure, respiration, urine secretion, temp. regulation, and basal metabolic rate. I possesses no chemotherapeutic or cytostatic effect, has no neg. influence on infections or on Ehrlich carcinoma in mice, it increases the effect of penicillin and Supracillin in the Aronson sepsis in mice, and does not affect the tuberculostatic action of isoniazid and streptomycin in the exptl. tuberculosis of the mouse.

~12 Citings

146. 1-Amino-2-anthraquinonecarboxylic acid amides

By Ebel, Friedrich; Randebrock, Rudolf
From No Corporate Source data available (1955), US 2717898 19550913, Language: Unavailable, Database: CAPLUS

See Brit. 731,008 (C.A. 50, 8741e).

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147. 1-Amino-2-anthraquinonecarboxylic acid amides

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Amino compds. (I) with 3,4-phthaloylisatoic acid anhydride (II) give the title compds. The I contain at least one N-H bond, e.g., amines, hydrazines, and amidines; when they are 1-aminoanthraquinones [which have an H_2N, HS, or HO (or halogen) ortho to the H_2N group], hydrazine, or amidines, the title compds. (III) may be converted to imidazoles, thiazoles, oxazoles, oxadiazoles, or hydroxypyrimidines, resp., by eliminating NH_3, H_2O, or H halide. Substituted II may also be used. E.g., II 3 and $PhNH_2$ 15 parts boiled 2 h. with stirring evolve CO_2 and yield the calcd. amt. of 1-amino-2-anthraquinonecarboxanilide, m. 271-2°; it gives a yellow-brown color in concd. H_2SO_4 and a red vat with alk. $Na_2S_2O_4$ soln. The following III were thus prepd. [the starting I, the substituent (if any) on the II used, the m.p. of III, and the colors formed by treating III with concd. H_2SO_4 and with alk. $Na_2S_2O_4$ given]: $MeNH_2$, 234°, -, -; iso-Bu_2NH, 131-2°, red changing to yellow, red; PhNHMe, 198°, yellow, red; $PhNH_2$, 4-O_2N, above 300°, yellow, wine-red; 1-amino-5-benzamidoanthraquinone, -, -, red-brown; 2-amino-3-hydroxyanthraquinone (IV), -, -, -; $PhC(:NH)NH_2$ [the III is named "1-aminoanthraquinone-2-carboxylic acid benzamidide" (V)], 217-18°, -, -; $N_2H_4.H_2O$, 258°, yellow, red; $N_2H_4.H_2O$, 4-MeO, 225°, yellow, orange; 1-amino-2-anthraquinonecarbonylhydrazine, above 300°, pale yellow, red; benzidine (the bis compd. is formed), above 300°, yellow-brown, brownish red; tolidine (the bis compd. is formed), above 300°, brown-yellow, brownish red; dianisidine (the bis compd. is formed), above 300°, yellow-brown, brownish red; the bis compd. prepd. from benzidine, 5-MeO, above 300°, yellow-brown, red; benzidine (the bis compd. is formed), 4-MeO, above 300°, above brown, red; tolidine (the bis compd. is formed), 4-MeO, 300°, brown, red; dianisidine (the bis compd. is formed), 4-MeO, above 300°, brown, red; tolidine (the bis compd. is formed), 4-Cl, 290-1°, yellow-brown, red-brown; 2,2'-diamino-4,4'-bithiazole (the bis compd. is formed), golden yellow, red-brown. When heated with p-$MeC_6H_4SO_3H$ in $C_6H_3Cl_3$, the III from II and IV gives 2-(1-amino-2-anthraquinonyl)anthraquinono-2',3':4,5-oxazole (cf. Brit. 298,545, C.A. 23, 3103). V boiled with Ac_2O gives 2-phenyl-4-hydroxy-7,8-phthaloylquinazoline, m. above 300°. 2,5-Bis(1-amino-2-anthraquiny)-1,3,4-oxadiazole is prepd. by heating II with N_2H_4 in $PhNO_2$, removing the solvent, and treating the product with $SOCl_2$. The colors obtained in dying cotton are described for some of these dyes.

~0 Citings

148. New synthesis of phthalimidoacetonitriles and a note on aminoacetonitrile sulfates

By Stephen, Henry
From Journal of the Chemical Society (1931), 871-5. Language: Unavailable, Database: CAPLUS, DOI:10.1039/JR9310000871

$C_6H_4(CO)_2O$ and $CH_2:NCH_2CN$, heated until the evolution of HCHO ceases, give a good yield of phthalimidoacetonitrile (I), m. 123-4°; I was also prepd. from K phthalimide and $ClCH_2CN$ and by dehydrating II with P_2O_5; 4-Cl deriv., m. 146.5°; 3-NO_2 deriv., yellow, m. 156°; 4-NO_2 deriv., yellow, m. 134-5°; 3,6-Cl_2 deriv., m. 174-5°; 3,6-Br_2 deriv., m. 228°; tetra-Cl deriv., m. 259° ($C_6H_4Me_2$ was used in its prepn.). Dissolving I in 80% H_2SO_4 gives phthalimidoacetamide (II), m. 257°; this also results by heating $C_6H_4(CO)_2O$ a few degrees above the m. p. and adding the HCl or H_2SO_4 salt of H_2NCH_2CN; soln. in cold NaOH and acidification gives phthalamic acid N-acetamide, m. 204-5°; above its m. p. it gives II. 4-Chlorophthalimidoacetic acid, formed from the nitrile in dil. HCl at 60° or from 4-$ClC_6H_3(CO)_2O$ and $H_2NCH_2CO_2H$, yellow, m. 205° (chloride, m. 93.5°; amide, m. 241°; Et ester, m. 95.5°); 3-NO_2 deriv., yellow, m. 208° (chloride, m. 119.5°; amide, m. 212°; Et ester, m. 77.5°); 4-NO_2 deriv., m. 193° (chloride, buff, m. 129°; amide, m. 214°; Et ester, m. 78°); 3,6-Cl_2 deriv., m. 243-4.5° (chloride, m. 135°; amide, m. 262-3°; Et ester, m. 193°); 3,6-Br_2 deriv., m. 239-42° (amide, m. 285-7°); tetra-Cl deriv., m. 298° (decompn.) (chloride, m. 209° Me ester, m. 181°; Et ester, m. 180.5°; amide, m. 294° or slowly heated, decomps. 285°. Naphthalimidoacetonitrile, pale yellow, m. 248° (decompn.); the acid m. 259-60°; chloride, pale yellow, m. 232°; amide, yellow, m. 319°. $CH_2:HNCH_2CN$ with concd. H_2SO_4 in EtOH with cooling give the monosulfate of H_2NCH_2CN, hygroscopic, m. 121°; Klages (Ber. 36, 1506 (1903)) gives 101°; Klages' method gives a mixt. of the 2 sulfates. Both sulfates react with $C_6H_4(CO)_2O$ and derivs. to give the corresponding amides.

~0 Citings

149. 3,4-Diaminophenylarsonic acid and some of its derivatives

By Lewis, W. Lee; Bent, H. E.
From Journal of the American Chemical Society (1926), 48, 949-57. Language: Unavailable, Database: CAPLUS, DOI:10.1021/ja01415a014

At 100-40° and 10 mm. pressure, over P_2O_5, the compds. $H_2NC_6H_4AsO_3H_2$, $HO_2CC_6H_4CONHC_6H_3(NH_2)AsO_3H_2$ and $CH_2(OCNH)_2C_6H_3AsO_3H_2$ lose H_2O; the Mg salts of these compds. do not lose H_2O under the same conditions, showing that the loss takes place from the arsonic group. 4-Propionylaminophenylarsonic acid, does not m. at 260° (32% yield). The following derivs. of $H_2NC_6H_4AsO_3H_2$ were prepd. by a modified Schotten-Baumann reaction: 4-Valeryl, 59% yield; 4-phenylacetyl, 81%; 3-amino-4-acetylamino, m. 265-7°, 74%; 3-amino-4-propionyl, m. 230-5°, 40%; 3,4-divaleryl, 44%; 3,4-dichloroacetyl, 49%; 3,4-diphenylacetyl, 62%; 3,4-dibenzoyl, 53%; 3,4-diphthalyl, 16.5%; 4-carbopropyloxy, 81%; 4-carbobutyloxy, 43%; 3,4-dicarbethoxy, m. 192-2.5°, 23%; 3,4-dicarbopropyloxy, decomps. 249-53°, 57%; 3,4-dicarbobutyloxy, decomps. 185-7°, 13.6%. Aminoarsanilic acid (I), and $ClCH_2CONH_2$ in NaOH give 62% of N-[phenyl-1-amino-4-arsonic acid]-glycine amide, light gray powder, darkens 215°, m. 234-41° (decompn.); using 25% more alkali or recrystn. of the amide in excess of alkali gives 1,2-dihydro-3-amino-6-arsonoquinoxaline (II), m. 226° NH_4 salt, decomps. 200°. I and $(COCl)_2$ give 32% of the 1,2-dihydro deriv. Bz deriv. of II, m. 234°; hydroxyethyl deriv., in 34% yield from II and $(C_2H_4)_2O$. I and $BrCH(CONH_2)_2$ give the 1,2-dihydro-2-formamide-3-amino deriv. in 17.2% yield. Aminotryparsamide and HCH(OH)OSONa give a ppt. during the 1st half hour which appears to be the arseno compd. with but 1 NH_2 group reacting; after 15 h., there results 75% of the arseno deriv. of sulfoxylated aminotryparsamide, light yellow powder, does not change up to 260°. N-[4-Arsonophenyl]-aminomalonamide, 57% yield. I and $CH_2(CO_2Et)_2$ in MeOH give 26% of malonylaminoarsanilic acid, light red, which loses H_2O at 139°. 4-Phthalylamino-3-aminophenylarsonic acid. in 15% yield from I and $C_6H_4(CO)_2O$; it seems to lose vol. up to 203-7°, then increase in vol. at 240-5° with no further change to 260°. 1,2-Di-[N,N-dimethylamino]-4-arsenobenzene, light pink, by refluxing I in MeOH-HCl.

~0 Citings

150. Action of diamines on naphthalic anhydride

By Bistrzycki, A.; Risi, J.
From Helvetica Chimica Acta (1925), 8, 810-20. Language: Unavailable, Database: CAPLUS

On 2 successive occasions 1,8-$C_{10}H_6(CO)_2O$ (I) suspended in boiling alc. and alc. $N_2H_4.H_2O$ gave 1,8-naphthalhydrazide, $C_{10}H_6(CONH)_2$ (II), leaflets or pale yellow needles, m. 254-5° (darkening), does not yield a benzal deriv. or combine with more I as $C_{10}H_8(CO)_2NNH_2$ (III) (Ostrogovich and Mihailescu, C. A. 6, 995) does; it m. 242° when mixed with III; di-Ac deriv., m. 214-5°. After several months further attempts to prep. II resulted only in the formation of III. I and $(CH_2NH_2)_2$, heated in boiling alc. only until crystals sep., give N-[8-carboxy-1-naphthoyl]-ethylenediamine (IV), $HO_2CC_{10}H_6CONHC_2H_4NH_3$, decomps. 196-7°, splits into its components in the higher boiling solvents, is partly sol. in dil. aq. $NaHCO_3$ or HCl, fluoresces more strongly in H_2SO_4 than does I; Pb salt, amorphous. At 230-40° for 10-15 min. IV yields 1,2-[1',8'-naphthoylene]-imidazole 4,5-dihydride $CH_{10}H_6.CO.N.CH_2.CH_3N:C$, greenish yellow, m. 179-80° (decompn.), fluoresces in H_2SO_4, stable to boiling alc. KOH. 8-Carboxy-1-naphthoyl-o-phenylenediamine (V), $HO_2CC_{10}H_6CONHC_6H_4NH_2$, yellow, gradually loses H_2O when heated, m. 236-8° (decompn.), partly sol. in dil. aq. $NaHCO_3$ or HCl, sol. in H_2SO_4 with an orange color; Ag salt, amorphous. Heated 10 min. at 150°, then gradually to 230-40°. V forms 1,2-[1',8'-naphthoylene]benzimidazole, $C_{10}H_6.CO.N.C_6H_4.N:C$, greenish yellow, m. 189°, sol. in H_2SO_4 with a greenish yellow color. Unlike the corresponding benzoylene compd. (B. and Cybulski, Ber. 25, 1990(1892)) it is not reduced by Zn dust in boiling HOAc. N-[1'-Amino-2'-naphthyl]-1,8-naphthalamidic acid (VI), almost colorless, m. 143-4° when rapidly heated, sol. in H_2SO_4 with a brownish orange-red color; Ag salt, flocks; 1,2-[1',8'-naphthoylene]-α-naphthimidazole, from VI, heated 15 min. at 230-40°, brownish orange, m. 238-9° (decompn.). To decide whether condensation in VI had actually occurred on the 2-NH_2 group of 1,2-$C_{10}H_6(NH_2)_2$, (VII) $C_6H_4(CO)_2O$ and VII were heated in boiling alc. or PhH, giving N-[1'-amino-2'-naphthyl]phthalamidic acid (VIII), decomps. ca. 205° (Ag salt, amorphous), converted by treatment with 12% HCl, drying on clay, and boiling under a reflux with AmONO and a few drops of 12% HCl into o-$HO_2CC_6H_4CONHC_{10}H_7$(β), dimorphic. At 270-80° for 15 min. VIII yields 1,2-[1',2'-benzoylene]-α-naphthimidazole, orange, m. 208°, sol. in H_2SO_4 with an orange color.

~2 Citings